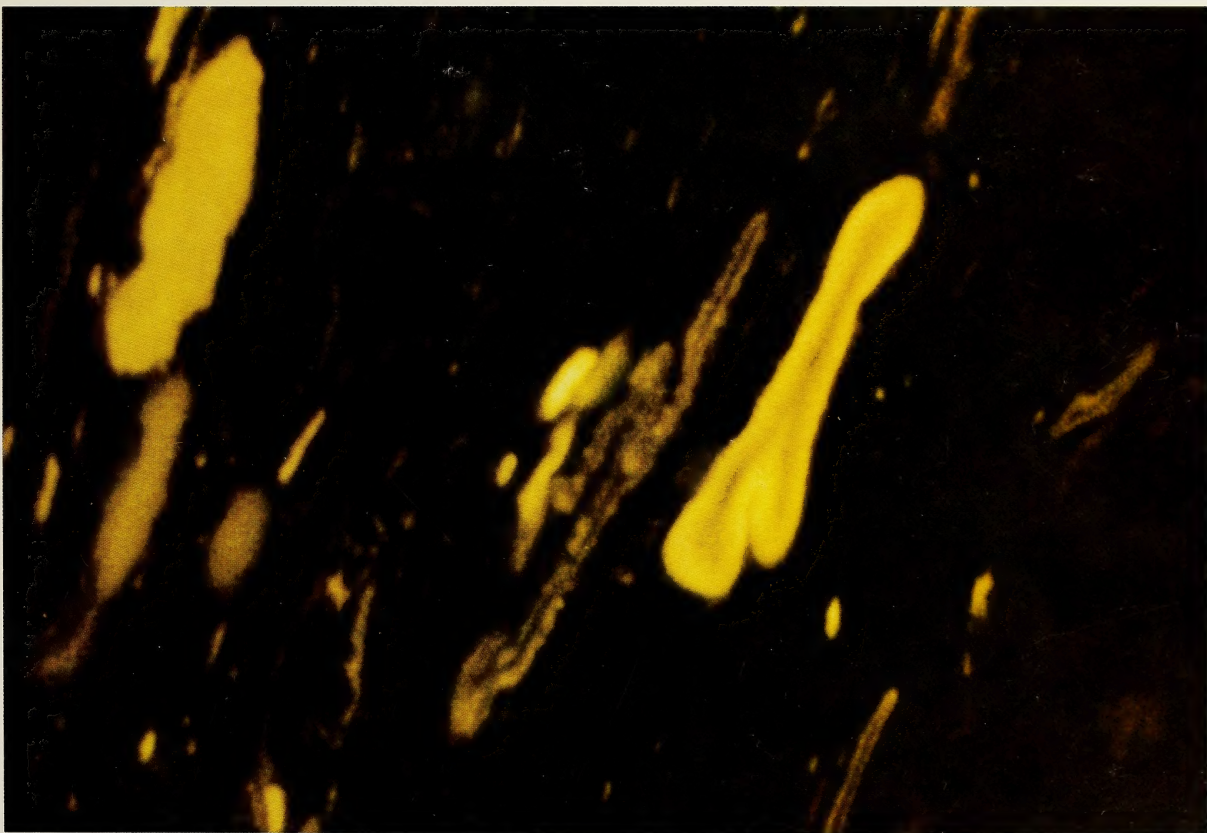


# Petrographic studies of coals from Alberta plains

S.Parkash



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S.Parkash

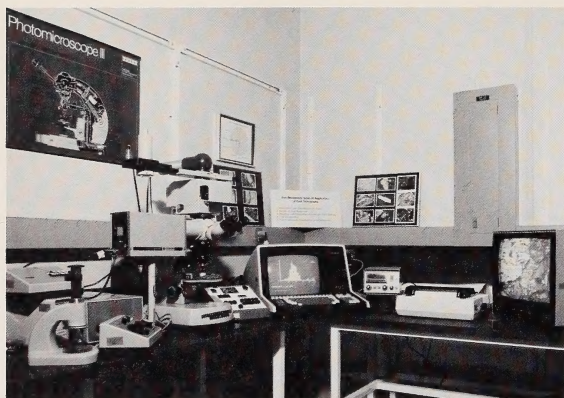


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## Definitions

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Coal petrology is the science concerned with the nature, origin, evolution and significance of coal as a rock material. Coal petrography is the subsience concerned with the macroscopic and microscopic description of coal materials and the practical use of compositional descriptions.

Macerals are fundamental ingredients of coal

analogous to minerals in other rocks.

Reflectance and florescence are optical properties of coal and are used to express rank or degree of metamorphism. The resulting data are used to interpret the history of a coal seam and to assess its technological behavior.

## Summary

Eleven *coal regions* in the Alberta plains, identified suitable as feedstock for on-site conversion by liquefaction and pyrolysis, were analyzed for their petrographic composition and rank determination by reflectance analysis. Four lignites, two from Saskatchewan and one each from Texas and North Dakota, were studied for comparison. Petrographically, the eleven subbituminous coals could be divided into two populations: one with high huminite and liptinite contents (90 percent) and one with substantially lower contents (70 to 75 percent). On a geological formation basis, the average huminite (vitrinite) content in coals from the Horseshoe Canyon Formation (88 percent) was higher compared to coals from the Paskapoo Formation (72 percent). The abundance of semifusinite in coals from the Paskapoo Formation (for example, Highvale and Ardley coals) suggested swamp conditions during the coalification which accelerated oxidation on the accumulating vegetation. The two American lignites showed huminite plus liptinite contents of less than 70 percent while the Saskatchewan lignites were somewhat higher. Reflectance measurements on the subbituminous coals showed a range of 0.36 to 0.47 with a reasonable correlation to the ASTM rank designations of these coals. Reflectivities determined on the lignites were generally lower.

To investigate the precise role of different organic coal components in technical processes, maceral concentrates from subbituminous and lignite coals were fractionated by the float-and-sink method using liquids differing in specific gravity. Lighter fractions showed enrichment of huminite and liptinite, but inertinite and mineral matter were in the heavier splits. The chemical composition of pure macerals computed from petrographic analyses of maceral concentrates showed that the huminite macerals were relatively rich in oxygen, whereas the liptinite macerals were relatively rich in hydrogen and the inertinite macerals were hydrogen deficient but relatively rich in carbon. To isolate relatively pure macerals from subbituminous coals, a density gradient centrifugation technique (DGC) was developed to obtain macerals of greater than 90 percent purity. A major limitation of the DGC

maceral separation procedure was the small amount of material obtained in a single DGC run. For maceral reactivity studies, where somewhat larger quantities were needed, multistage float-and-sink tests followed by density gradient centrifugation were suggested for isolating pure macerals from tests in microreactors. To obtain larger quantities of macerals (a few grams), a compromise between quality and quantity was the solution. An examination of the relationship between liquefaction yields and petrographic composition showed a rough positive correlation with huminitic-liptinitic content.

In order to follow the progressive alteration and resolidification of coal components during liquefaction, liquefied coal residual materials from both batch autoclave tests and continuous runs were examined. This examination was based on three main categories: relatively unaltered organic components, thermally altered components (vitroplast, cenospheres, semicoke and granular residue), and inorganic components. Microscopic examinations of liquefaction residues from optimum conditions suggested total conversion of liptinite, extensive conversion of huminite and possible partial conversion and reactivity of the semifusinite maceral of the inertinite group. An investigation of coal maceral morphology changes upon progressive hydrol liquefaction provided some new insights into the role of different macerals in coal liquefaction in a semiquantitative manner. Because of the extremely complex catalytic role of mineral matter in coal, it was not possible to predict the liquefaction of a coal from its maceral composition alone. It was not confirmed whether comparative studies of maceral composition and rank with those of the relevant hydrogenation residues should enable conversion rates and efficiencies to be assessed for the various coals. The assessment of coal hydrogenation performance using microscope techniques might not be very efficient. It appeared essential to pool results from both chemical and microscopic investigations to derive a better understanding of the hydrol liquefaction process.

## Background

In the 1979 long range plan on major expansion of research on coal, the Alberta Research Council recognized the importance of coal petrology to strengthen technical competence in supporting research and development for major thrusts in coal liquefaction and coal pyrolysis. The Alberta Research Council has had a modest but internationally significant program of coal research throughout its history; however, facilities for coal petrography in terms of equipment and expertise were missing until 1980. With the implementation of a new coal research program, funded jointly by Alberta Energy and Natural Resources and the Alberta Research Council, it was

decided to establish a petrographic support capability. It took almost two years to build updated petrography laboratories and to learn the state of the art with the cooperation of the Institute of Sedimentary and Petroleum Geology, Calgary, Alberta. The Alberta Research Council now has world class facilities in coal petrography and has been recognized by the International Committee on Coal Petrology.

To set objectives and the scope of this work, six applications of coal petrography were recognized:

1. description and classification of coals
2. survey of coal resources
3. correlation of coal seams and interpretation of



- geological structure
- 4. blending and preparation of coals for coke making
- 5. coal beneficiation
- 6. coal liquefaction, pyrolysis and combustion

In order to further the objectives of the coal research carried out within the Coal Research Department, the studies focussed primarily on the first and last applications. The goal of this program is to investigate the

petrography of Alberta subbituminous coals with reference to liquefaction and pyrolysis potential. The ultimate aim is to predict the yield of liquid and gaseous products, starting from the given rank of the coal and a given maceral composition. This report deals specifically with principles and applications of subbituminous coal petrography.

## Introduction

Petrography classifies coal by its organic and mineral constituents. Although well-established for bituminous coals, it requires further development for subbituminous coals. In 1980 the Alberta Research Council initiated a program for evaluating the subbituminous coals of the Alberta plains for pyrolysis, combustion and liquefaction behavior. Unlike their chemical composition, the petrographic characteristics of these coals were almost unknown. It was decided to include petrography as part of the analytical procedures because of its possible application in explaining, improving and controlling the technological behavior of coal, specifically, in this instance, liquefaction.

The essence of the petrographic approach to the study of coal composition is the idea that coal is composed of a number of distinct components, called macerals, each of which has a distinct set of physical and chemical properties. Depending on their quantitative participation and their association, macerals control the chemical, physical and technological properties of a coal of a given rank (ICCP, 1983).

The basic premise of the applications of coal petrography is that maceral analysis and reflectance analysis characterize a coal so well that they can be used to predict the behavior of that coal in any technological process of interest.

To establish a petrographic support capability for the

liquefaction and pyrolysis programs, the present investigations focus on the following technical objectives:

1. to establish a maceral classification for subbituminous coals from the Alberta plains
2. to scan Alberta subbituminous coals for their petrographic composition
3. to develop and evaluate automated reflectance microscopy for coal rank determination
4. to standardize and implement the procedures for separation of macerals and to investigate their liquefaction behavior
5. to develop a classification system for subbituminous coal liquefaction residual materials

The ultimate aim of these studies is to predict the yield of liquid and gaseous products from a given coal based on its maceral composition and rank.

At present the relationship between coal characteristics and liquefaction is known in general terms (Given *et al.*, 1975; Barron *et al.*, 1979; Diessel, 1979), although not for Alberta plains coal. The present study attempts to explore this relationship for Alberta subbituminous coals by a systematic petrographic study of several coal deposits, coupled with an examination of the solid residues resulting from liquefaction tests.

## Maceral nomenclature - subbituminous coal

From the petrographer's viewpoint, coal is composed of phytogenetic macerals, (Spackmann, 1958). Macerals are the optically homogeneous organic materials in coal which are microscopically recognizable as individual coal constituents. They are identified and classified on the basis of their morphology, color or level of reflectivity, source material and nature of formation. There is a much greater degree of petrographic heterogeneity in subbituminous coals than in coals of higher rank. This is to be expected since the vegetal materials have been less altered, and it is thus possible to detect more readily the variations in the nature of plant tissues and peat swamp. Because of this fact and because the nature and properties of some of the materials are different from those of the macerals of coals of higher

rank, it was essential to establish a maceral nomenclature for low rank coals, namely, subbituminous coals and lignites. Table 1 summarizes the subbituminous coal maceral groups.

The organic or nonmineral part of coal consists of three major groups of macerals:

1. vitrinite ("Huminite" is restricted to low rank coals -lignites and subbituminous. In discussing the results of the present study the term huminite will be used.)
2. liptinite (exinite)
3. inertinite

Basically, the huminite group of this classification is equivalent to and the precursor of the vitrinite group of bituminous coals. Macerals of the liptinite and iner-



tinite groups are used under the same names as in bituminous coals.

Representative photomicrographs of macerals are shown in plates 1-4. Each group includes a series of macerals that either have a similar origin or the same mode of conversion from the original plant material to

coal. The three maceral groups are, to a certain degree, characterized by their elemental composition. On comparing groups of the same rank, huminite (vitrinite) contains relatively more oxygen, the liptinite more hydrogen and the inertinite more carbon.

**Table 1.** Maceral nomenclature — subbituminous coal\*

Group maceral	Maceral subgroup	Maceral	Source	
Huminite	Humotelinite	Textinite	Ungelified cell wall material	
		Uliminite	texto-ulminite	Gelified plant tissues with recognizable cell structure
			eu-ulminite	
	Humodetrinite	Attrinite	Humic Detritus	
		Densinite		
	Humocollinite	Gelinite	Formless humic gels	
Corpohuminite		Secondary colloidal gel excretions, and primary cell infillings (tannins)		
Liptinite	Sporinite	Spore exines		
	Cutinite	Cuticles		
	Resinite	Resins		
	Suberinite	Cork tissues		
	Alginite	Algae		
	Liptodetrinite	Fine particles of liptinite		
Inertinite	Fusinite	As in hard coals		
	Semifusinite			
	Macrinite			
	Sclerotinite			
	Inertodetrinite	Fine particles of inertinite		

\* Modified after *Summary of the macerals of brown coal* in short course on the Theory and Practice of Coal Petrology, W. Spackman and A. Davis, Pennsylvania State University, U.S.A., 1981.

## Description of macerals

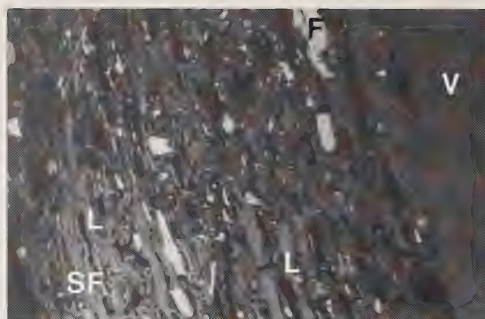
Huminite (vitrinite) is the most abundant and most important maceral in coals, accounting for, in most instances, more than 70-80 percent of any given coal bed. It is derived mainly from wood and bark. In reflected light, it appears dark gray, light gray or white with a progressive increase in reflectance as rank increases. Distinction between the macerals in the huminite group results mainly from different degrees

of gelification.

Generally, textinite produces a high tar yield and much gas on low-temperature carbonization. This is because of its frequent high cellulose or resin content. Textinite occurs only rarely in Alberta plains coal. Ulminite yields little tar and gas and more coke relative to textinite on low-temperature carbonization. The technological properties of ulminite depend upon its degree of gelification.



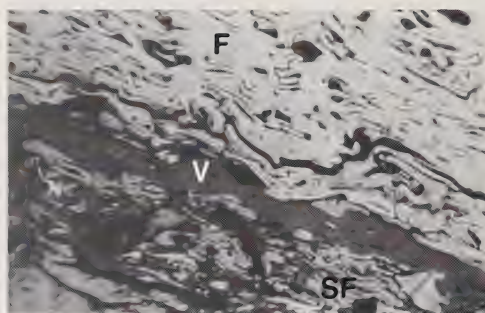
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2



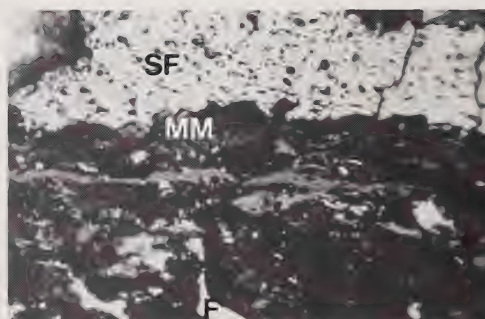
3



4



5



6

0 —  $\mu\text{m}$  — 100

**Plate 1.** Photomicrographs of Smoky Tower coal:

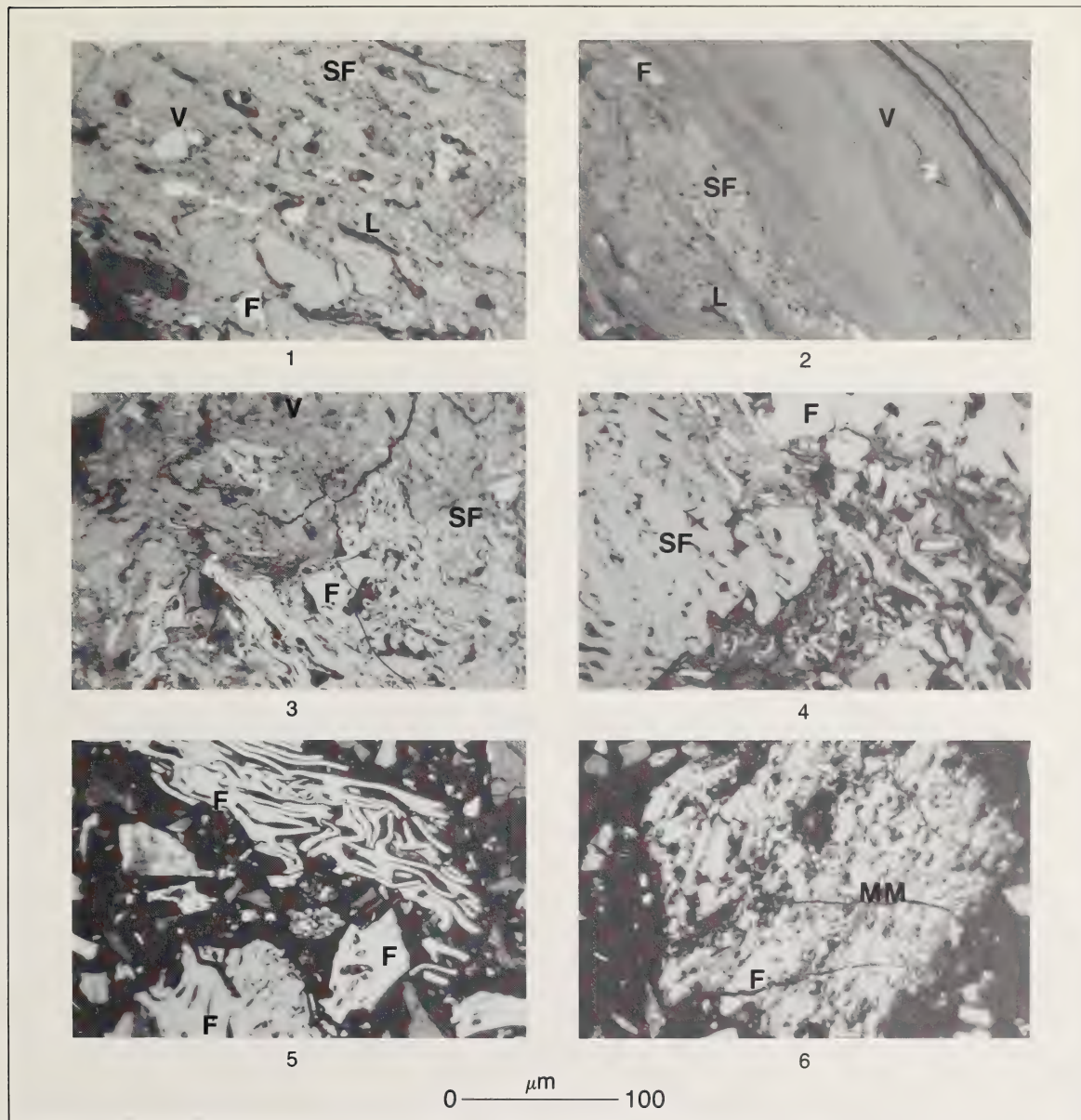
(1) composite sample, (2) density fraction  $< 1.35$ , (3) density fraction  $1.35-1.45$ , (4) density fraction  $1.45-1.55$ , (5) density fraction  $1.55-1.60$ , (6) density fraction  $> 1.60$ , showing vitrinite (V), lignite (L), semifusinite (SF), fusinite (F) and mineral matter (MM)



Humodetrinite yields little tar in low-temperature carbonization. It gives relatively poor yields of bitumen on extraction and is oxidized relatively easily.

Humocollinite may constitute a major portion of the huminite group maceral which originates from humification and subsequent metamorphosis of cell-wall materials from wood or cortex tissue. It is very susceptible to weathering and has a lower calorific value than the average for subbituminous coal.

The macerals of the liptinite group are derived from a variety of parent materials, such as the exines of spores and pollen, cuticles, resins, waxes, and algae. Macerals of this group share the common characteristic of having the highest hydrogen content of all coal constituents (Ting, 1972). Liptinites are dark gray to gray in reflected light in lower rank coals (lignite to high volatile bituminous). In higher rank coals, they become indistinguishable from huminite because of a



**Plate 2.** Photomicrographs of Highvale coal:

(1) composite sample, (2) density fraction < 1.35, (3) density fraction 1.35-1.45, (4) density fraction 1.45-1.55, (5) density fraction 1.55-1.60, (6) density fraction > 1.60, showing vitrinite (V), liptinite (L), semifusinite (SF), fusinite (F) and mineral matter (MM)

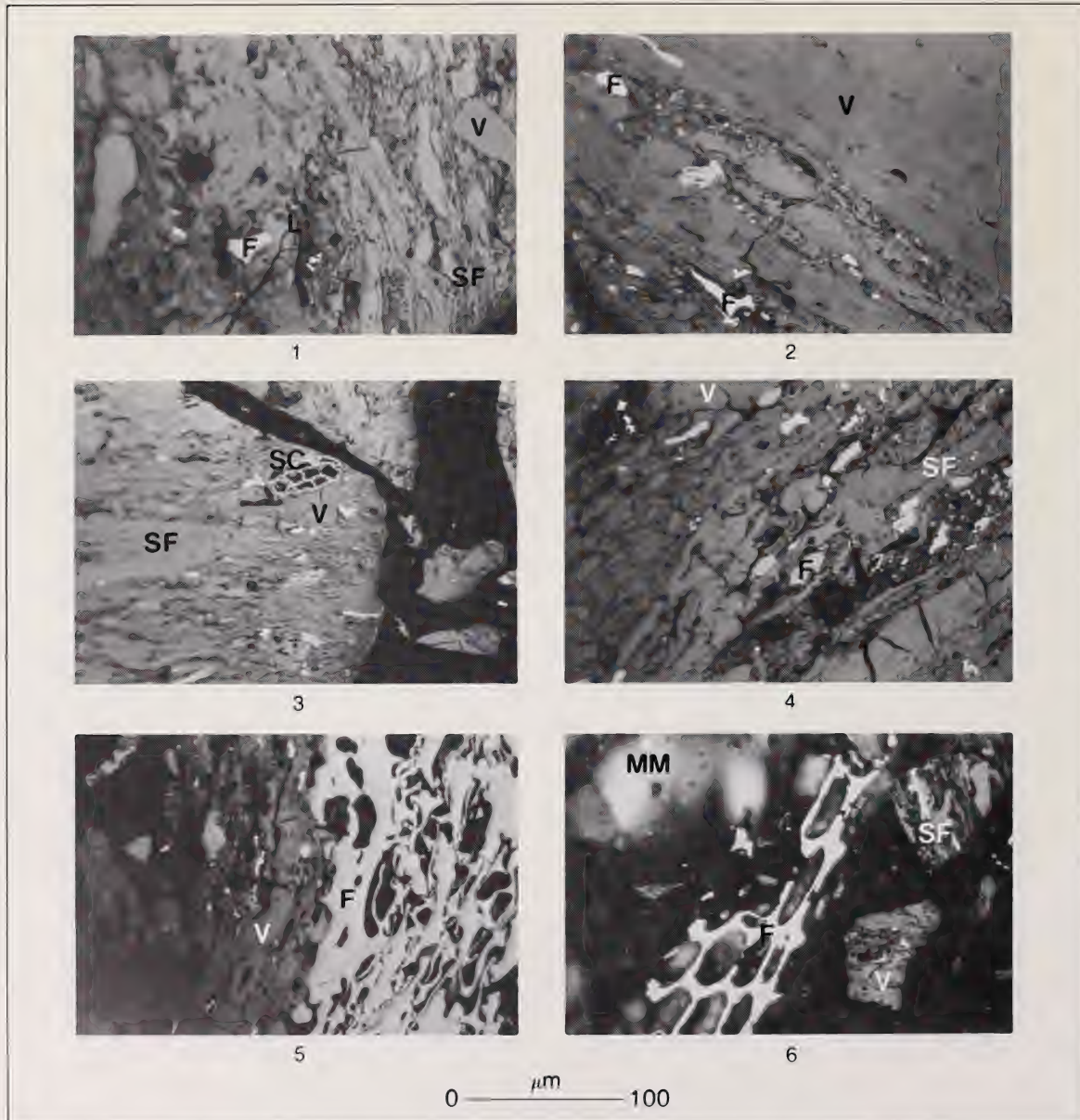


rapid increase in reflectance. To identify the macerals of liptinite, florescent light is usually recommended, especially in low rank coals.

Inertinites are the group of macerals that are bright in reflected light. Inertinite derives its name from the fact that it is inert or semi-inert during the normal carbonization process. Depending on their shape, size, degree of preservation of cellular structure and reflectance, inertinites are subdivided into fusinite,

semifusinite, inertodetrinite, macrinite, micrinite and sclerotinite.

The fusinite maceral is the richest in carbon of all the constituents of coal. In general, its proportion is low and does not exceed a small percentage. In reflectd light it is yellowish-white to white and the reflectance is high. It is not true that the reflectance of fusinite is always the highest of all macerals (Alpern and Lemos De Sausa, 1970). The density of fusinite, about 1.5



**Plate 3.** Photomicrographs of Vesta coal:

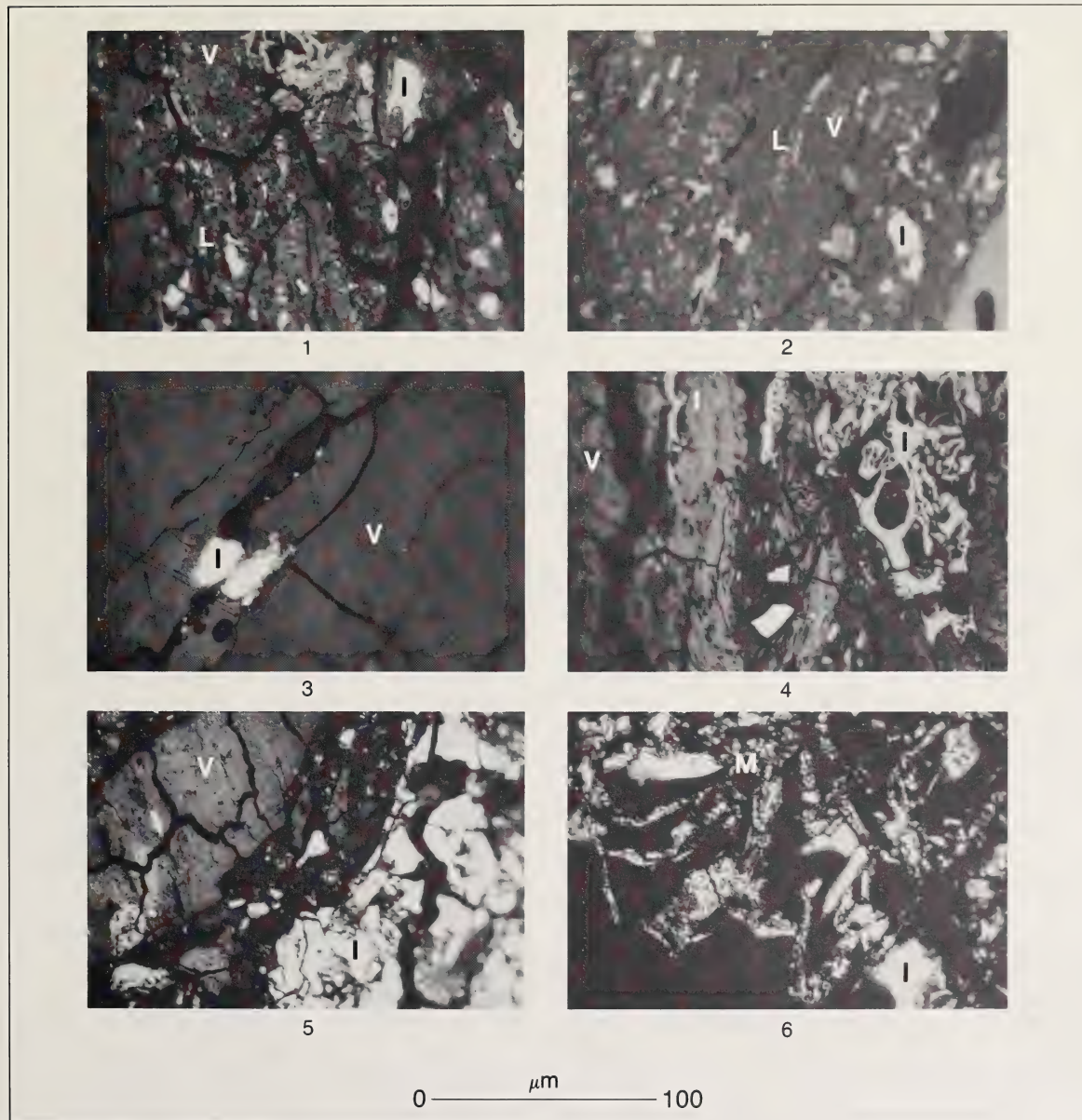
(1) composite sample, (2) density fraction  $> 1.35$ , (3) density fraction 1.35-1.45, (4) density fraction 1.45-1.55, (5) density fraction 1.55-1.60, (6) density fraction  $< 1.60$ , showing vitrinite (V), liptinite (L), semifusinite (SF), fusinite (F) and mineral matter (MM), sclerotinite (SC)

$\text{gcm}^{-3}$ , exceeds that of huminite and liptinite. During carbonization, the fusinite does not fuse and its physico-chemical properties in general do not vary much with increasing rank.

The semifusinite maceral is the transitional stage between fusinite and ulminite (huminite group maceral); the semifusinite maceral always shows reflectance intermediate between huminite and fusinite and is white or light gray. The physical and

chemical properties of semifusinite vary widely, ranging from those of huminite to those of fusinite, according to the degree of coalification. Semifusinite is richer in carbon and poorer in hydrogen than huminite, but poorer in carbon and richer in hydrogen than fusinite. The density varies from about 1.35 to 1.45  $\text{gcm}^{-3}$ .

Inertodetrinite is made up of strongly reflecting fine particles (normally less than 30  $\mu\text{m}$ ) and is believed to



**Plate 4.** Photomicrographs of North Dakota lignite:

(1) composite sample, (2) density fraction < 1.35, (3) density fraction 1.35-1.45, (4) density fraction 1.45-1.55, (5) density fraction 1.55-1.60, (6) density fraction > 1.60, showing vitrinite (V), liptinite (L), inertinite (I) and mineral matter (MM)



derive from fragmented fusinite and semifusinite.

Macrinite is a more or less amorphous, nongranular groundmass of high reflectance and is a relatively rare maceral. Micrinite is characterized by the rounded shape, the very small size (one  $\mu\text{m}$ ) of its grains and higher reflectance than the associated huminite.

Sclerotinite is recognized as fungal remains. The reflectance is commonly high and it is assumed that it contains much carbon and little hydrogen. The density of sclerotinite from soft subbituminous coals varies from 1.6 to 2.0  $\text{gcm}^{-3}$ .

The chemical and physical properties of the macerals (for example, elemental composition, moisture content, hardness, density and petrographic features) differ widely and are also subject to changes in the course of diagenesis and coalification. These differences in chemical and physical properties are reflected in the technological behavior of the macerals.

Table 2 gives, in a simplified manner, the technological behavior of macerals usually reported for coals up to the rank of low volatile bituminous (Bustin *et al.*, 1983).

**Table 2.** Technological behavior of macerals

Maceral group	Technological behavior		
	Chemistry	Carbonization	Liquefaction
Huminite	Intermediate hydrogen content and volatiles	Principal reactive constituent in coking	Susceptible to liquefaction
Liptinite	Higher hydrogen content and volatiles; more aliphatic	Reactive during carbonization	Very susceptible to liquefaction
Inertinite	Lower hydrogen content and volatiles; more aromatic	Inert during carbonization	Resistant to liquefaction

## Petrographic analysis of subbituminous coals

In 1980 the Alberta Research Council and Alberta Energy and Natural Resources recognized that under the conditions foreseeable for the near future, the preferred feedstock for on-site coal conversion in Alberta should be subbituminous coals from the plains region. Accordingly, 11 coal regions in the Alberta plains (shown on the map, figure 1) were selected for systematic scanning for their liquefaction and pyrolysis behavior. In order to investigate the compositional properties of Alberta coals and their liquefaction potential, these coals from the 11 regions were analyzed for their petrographic composition and rank determination by reflectance analysis.

All of the coal deposits tested (except the Buffalo Hill Region - Smoky Tower) happen to be subbituminous B or subbituminous C in rank (ASTM).

Geologically, all "deposits" referred to in this report are part of a single enormous wedge of coal-measures (with a few relatively thin marine bands included) that blankets the central and southern parts of Alberta (Campbell and du Plessis, 1983). Although the deposits occur at three distinct horizons within the wedge, they are so closely spaced in age (latest Cretaceous to earliest Paleocene) that they may be considered almost synchronous. The three horizons are: Oldman and Foremost Formations (Early Late

Cretaceous age), Horseshoe Canyon Formation (Late Cretaceous age), Scollard Member of Paskapoo Formation (Latest Cretaceous or Cretaceous-Paleocene boundary age).

Homogenized bulk samples representative of the recoverable coal deposits from the respective regions were obtained by a bucket auger drill rig. Samples were stored in sealed barrels at low temperatures to minimize oxidation. In addition to the 11 subbituminous coals, two American lignites (from Texas and North Dakota) and two Saskatchewan lignites were analyzed for comparison.

## Sample preparation and analytical methods

### Preparation

For the reflected light examination of the coals under study, specimens were prepared as briquets or pellets or crushed coal using an epoxy resin as the bonding medium. The crushed coal (less than 0.8 mm, that is, -20 mesh) was thoroughly mixed with the resin in the volumetric ratio of four parts coal to one part resin. This mixture was placed in a cylindrical plastic mold 2.54 cm in diameter and allowed to cure overnight. After removal from the mold, the pellets were ground



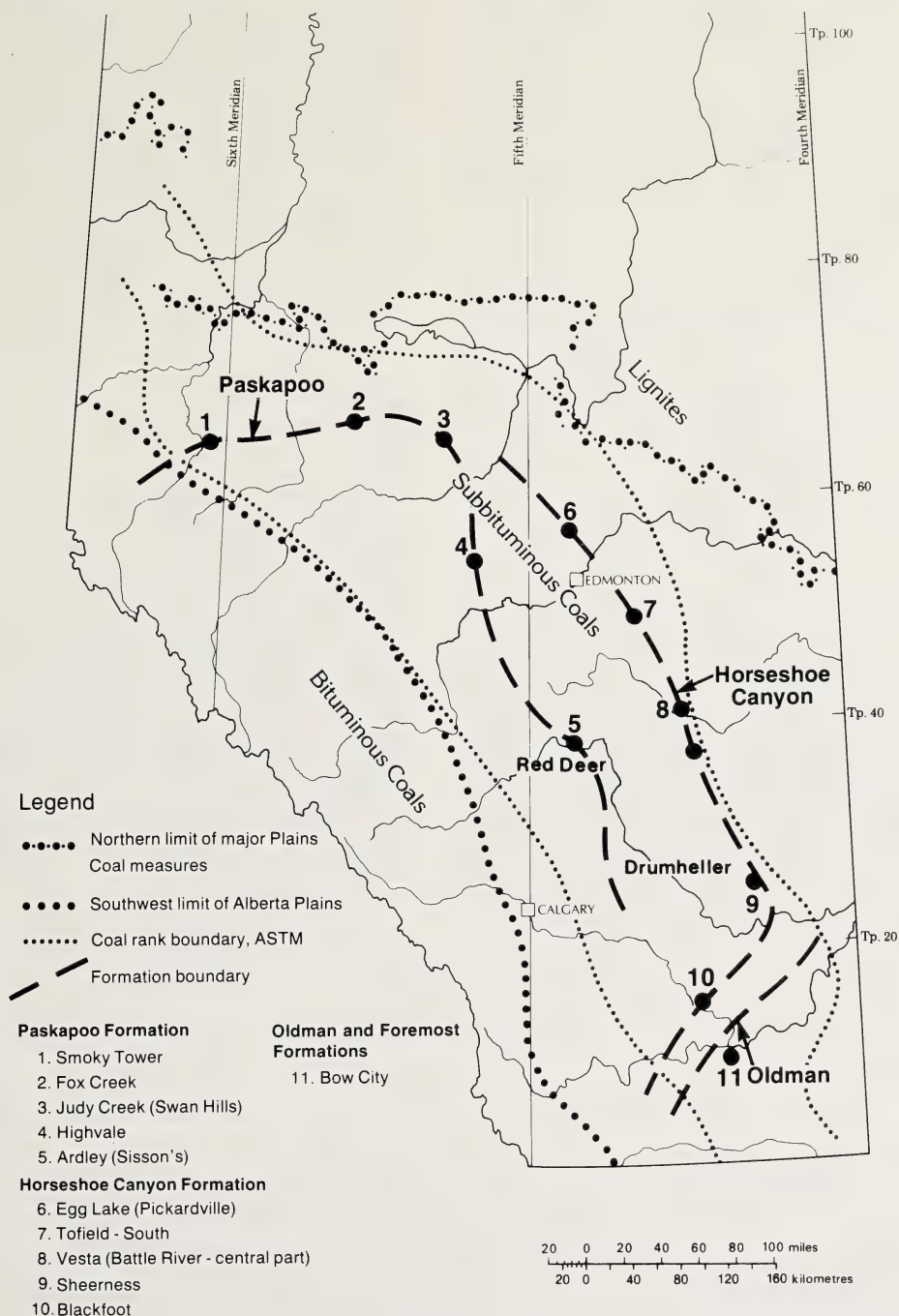


Figure 1. Location of tested coals

and polished with the Buehler Automet device. For detailed procedures on sample preparation, refer to ASTM Standard D-2797-72 (1984).

The reflectance values of some coals can be significantly affected by moisture content (Harrison, 1965, 1966). Consequently, pellets should be kept in a desiccator for 15 hours prior to measurement. In the case, however, of low rank coals, which tend to slack under dry condition, care must be taken to avoid excessive desiccation (Davis, 1978).

### Reflectance measurement

Reflectance, an index of the degree of metamorphism of coal, is defined as the proportion of normally incident light reflected by a plane, polished surface of the substance under consideration. Reflectance is usually expressed as a percentage, so that reflectance of 1.00 percent for huminite indicates that only proportion of the light incident on the coal surface is reflected under the conditions of measurement.

Measurement of the reflectance of the coal maceral huminite has become a routine procedure in the characterization of coal rank. Rank determination is performed by measuring either maximum reflectance in polarized reflected light or random (average) reflectance, usually in nonpolarized reflected light. The choice between the two techniques depends upon individuals. Measuring maximum reflectance requires that the object stage be rotated until a maximum value of reflectance is recorded.

When using random reflectance, the time consumed in rotation of the microscope stage is avoided and the measurement avoids error due to lack of centering and change in focus on rotation (Davis, 1978).

The automated, computerized Zonax system coupled with the Zeiss reflectance microscope were used for rank determination by measuring random

reflectance of huminite. Reflectances reported are mean values of at least 100 measurements per sample. Procedures for the measurement of reflectance are described in ASTM Standard D-2797-72 (1984). Reflectance values corresponding roughly to ASTM rank boundaries (Davis, 1978; Stach *et al.*, 1982) are given in table 3. Note the overlap between subbituminous A and high volatile bituminous B and C.

### Procedure of maceral analysis

In routine petrographic analysis, the relative proportions of macerals are obtained by the point count method, reported as volume percent. Within each field of view, the maceral under each intersection of the eye piece reticule is identified and counted.

In the present study about 900 points per sample were identified; the reported maceral contents are averages of three analyses by three operators on the same polished surface. The following macerals were reported: huminite, total liptinite, fusinite, semifusinite and other inertinite.

In recent years, florescent-light microscopy has proven very useful for studies of coal and holds great potential for routine petrographic analyses. In the rank range peat to subbituminous coal, liptinite exhibits a florescence of high intensity (Jacob, 1964). In the same rank range, not only the liptinite, but also some macerals of the huminite group exhibit florescence. In contrast, most macerals for the vitrinite group in bituminous coal do not floresce (Radke *et al.*, 1980). In general, macerals of the inertinite group do not floresce, although recently Koenigler reported the occurrence of florescing semifusinite in British Columbia coals (1980).

In the petrographic analysis of subbituminous coals, florescent-light microscopy was used to identify and quantify liptinic macerals.

**Table 3.** Relationship between coal rank and approximate reflectance limits\*

Coal rank		Percent reflectance	
Lignite		0.27-0.38	
Subbituminous	C	0.38-0.43	Random reflectance
	B	0.43-0.48	
	A	0.48-0.67	
High volatile bituminous	C	0.47-0.57	Maximum reflectance
	B	0.57-0.72	
	A	0.71-1.10	
Medium volatile bituminous		1.10-1.50	
Low volatile bituminous		1.50-2.05	
Semianthracite		2.05-3.00 (approx.)	
Anthracite		3.00 (approx.)	

\*From Davis (1978) and Stach *et al.* (1982).

## Results and discussion

### Petrographic analysis of subbituminous and lignite coals

The petrographic composition of the 11 subbituminous coals and four lignites is given in table 4. Photomicrographs representative of compositional association are included in plates 1-4. Plate 5 shows the macerals of the liptinite group in subbituminous coals after blue-light (fluorescence) excitation ( $\lambda = 450 - 490$  nm) indicating the presence of sporinite, cutinite and resinite.

Table 4 shows that, in terms of huminite content, the subbituminous coals consist of two populations. In one of these the huminite content exceeds 90 percent and,

together with liptinite, contains over 95 percent reactive material. The second set of samples contains lower contents of huminite with most values ranging between 70 and 75 percent. On the basis of geological formation, the average of the huminite content in coals from the Horseshoe Canyon Formation (88 percent) is higher in comparison to coals (72 percent) from the Paskapoo Formation (table 5). It is likely that the enrichment in huminite content is the result of washings accompanied by the flow of deposits away from the mountains during their coalification. The abundance of semifusinite in coals from the Paskapoo Formation (for example 22 percent in Highvale coal and 19 percent in Ardley coal) suggests swamp conditions

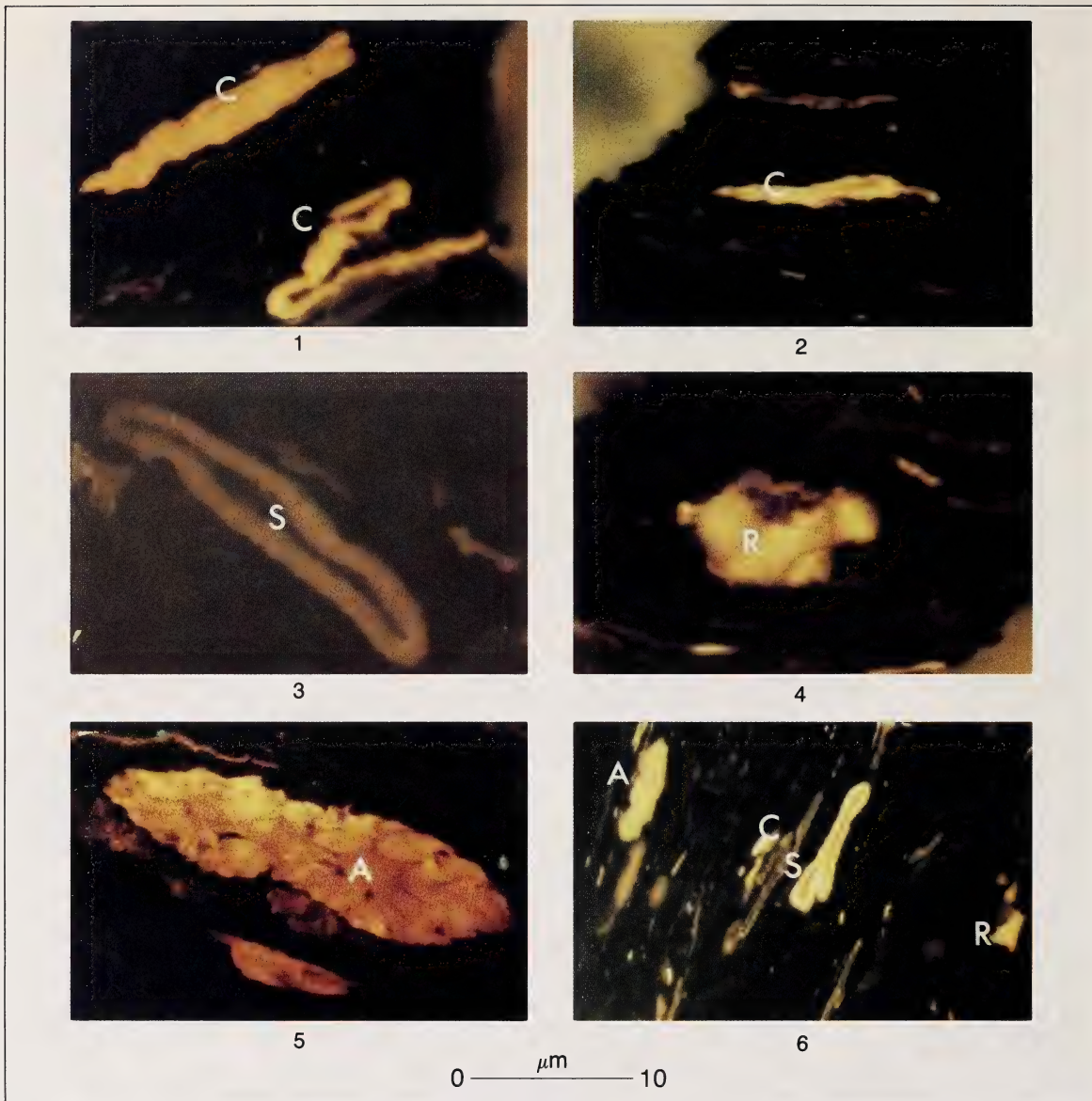
**Table 4.** Petrographic composition of subbituminous and lignite coals

Coal	Volume percent (mmfb)					Huminite random reflectance (percent)
	Huminite	Liptinite	Semi-fusinite	Fusinite	Other inertinites	
<i>Paskapoo Formation</i>						
1. Smoky Tower	70.0	6.4	8.6	4.3	10.7	0.47
2. Fox Creek	82.6	1.5	9.5	4.5	1.9	0.43
3. Judy Creek	72.4	5.3	8.1	9.1	5.1	0.38
4. Highvale	74.6	2.1	22.2	1.1	—	0.41
5. Ardley	59.5	9.0	18.9	7.5	5.1	0.45
<i>Horseshoe Canyon Formation</i>						
6. Egg Lake	92.4	4.2	0.8	0.4	2.2	0.42
7. Tofield	92.0	4.0	2.0	1.0	1.0	0.42
8. Vesta	73.3	0.4	8.9	3.3	14.1	0.36
9. Sheerness	92.7	5.3	1.2	0.8	—	0.47
10. Blackfoot	91.4	1.7	1.7	1.3	3.9	0.47
<i>Oldman Formation</i>						
11. Bow City	82.9	3.4	3.7	3.4	6.6	0.45
<i>Lignites</i>						
A Klimax, Sask.	83.1	0.4	3.9	1.8	10.8	0.36
B Bienfait, Sask.	69.0	13.0	9.0	2.0	7.0	0.36
C North Dakota	60.3	5.3	22.1	12.3	nil	0.24
D Texas	60.9	7.6	10.9	10.5	10.1	0.33

**Table 5.** Comparison between subbituminous coals from two geological formations for huminite content

% Huminite (mmfb)	
Paskapoo formation	Horseshoe formation
70.0	92.4
82.6	92.0
72.4	73.3
74.6	92.7
59.5	91.4
Average	88.4
71.8	





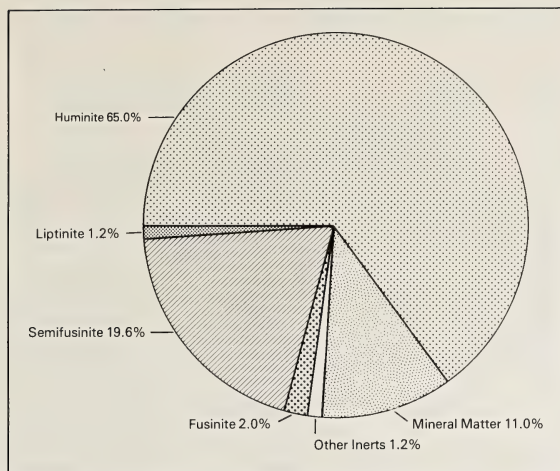
**Plate 5.** Photomicrographs (florescence mode) of liptinitic macerals: cutinite (C), sporinite (S), resinite (R), alginite (A).

during their coalification, which are conducive to accelerated oxidation and/or bacterial and fungal attack on the accumulating vegetal matter (Cameron and Kalkreuth, 1982).

Fires in or on the periphery of these swamps may have produced abnormal amounts of charred plant material now occurring in the coal as various forms of inertinite (Goodarzi, 1985). Alberta subbituminous coals are comparatively poorer in liptinite macerals and comparatively less abundant inertinite submaceral, the low density micrinite, is distributed uniformly. Maceral distribution in a subbituminous

(Highvale) coal is shown in figure 2.

Like the Saskatchewan coal from Bienfait, the two American lignites are relatively low in huminite, with values around 60 percent. Table 4 shows that the Texas lignite is richer in liptinite but poorer in semifusinite than the North Dakota material and the Texas coal contains considerable amounts of other inertinite macerals, apart from fusinite and semifusinite. Among the lignites, the sample from the Klimax Mine in Saskatchewan is highest in huminite with 83 percent. The highest liptinite content is in the Bienfait sample.



**Figure 2.** Maceral distribution in Highvale (subbituminous B) coal

The petrographic analysis of coal (especially of subbituminous and lignite coals) is a subjective technique, involving a certain amount of personal judgment. To show how the analyses made by different operators vary, table 6 gives the maceral composition for Smoky Tower (subbituminous A) coal provided by five operators. For all the subbituminous and lignite coals, the results given in this report seldom differed more than five percent.

Table 7 gives the detailed petrographic composition of two handpicked vitrain (huminitic) samples from the Vesta Mine (Horseshoe Canyon Formation). The huminite group is largely composed of eu-ulminite (greater than 65 percent), corpohuminites and humodetrinite. Texinite occurs only rarely. Other workers also report that North American coals usually do not contain texinite (Davis and Spackman, 1964).

The macerals of coal vary widely in their chemical composition and physical properties (Zwietering and van Krevelen, 1954). With increasing rank, these variations diminish. To check on the relationship between maceral composition (table 4) and chemical composition (table 8) in the subbituminous coals and lignites,

the volume percent of huminite plus liptinite and fusinite plus semifusinite versus H/C and O/C atomic ratios were plotted in figures 3 and 4. The data indicate that while there is a loose correlation between huminite plus liptinite contents and H/C atomic ratios, the concentration of fusinite plus semifusinite does not correlate in any manner to the chemical composition of subbituminous coals and lignites under investigation. These observations suggest that huminite and liptinite macerals, which account for more than 75 percent of the total organic macerals, might be relatively good diagnostic indicators for the chemical parameters (such as C and H) of subbituminous coals and lignites, provided that due attention is paid to the total petrographic composition and mineral matter content of the coals under investigation. The hydrogen content of the inertinite macerals, such as semifusinite, may be highly variable, as is the final disposition of hydrogen from the water of constitution in the clay minerals. The anomalous position of four samples (Smoky Tower and Vesta subbituminous coals and the two American lignites) in figure 3 may reflect such uncertainties in hydrogen content. In this connection it may be significant that three of these four samples have the highest combined content of mineral matter and inertinite of the 15 samples studied. This problem will be referred to again in the discussion of the chemical and petrographic properties of float-sink fractions.

#### Petrographic composition variations for a coal deposit within a seam

Tables 9 to 11 show variations in the petrographic composition for a coal deposit within a seam. Data for eight samples of Highvale Coal, seven samples of Ardley coal (both of Paskapoo Formation) and five samples of Vesta coal (Horseshoe Canyon Formation) indicate the degree of heterogeneity in coals from different locations. Variations in the petrographic composition for coal collected from five different core-holes in the Ardley region (W-4 meridian) are shown in table 12.

#### Petrographic composition of oxidized coals

The petrology of oxidized coals is studied to gain an understanding of the cause of spontaneous combustion (Chandra, 1962). To explore the morphological

**Table 6.** Comparison of petrographic analyses of a subbituminous coal\* (Smoky Tower — subbituminous A) conducted by five operators

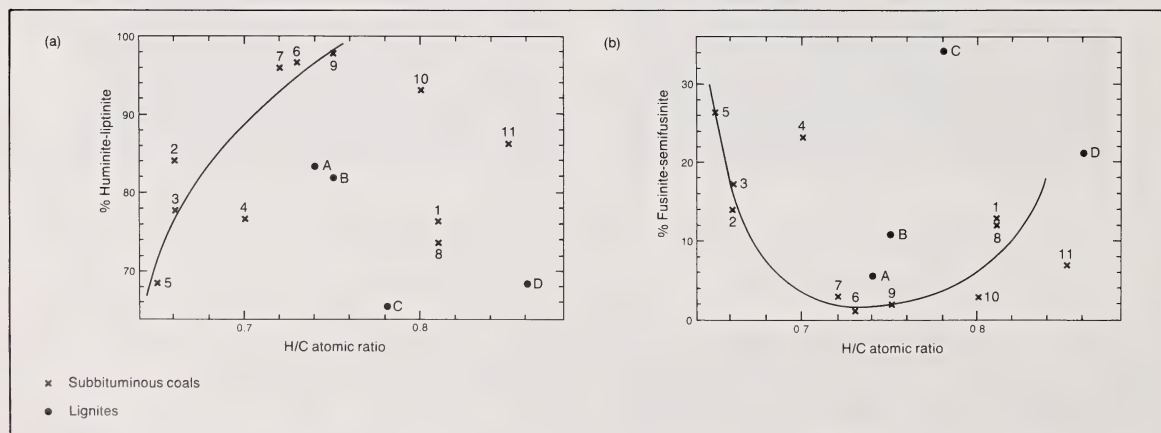
Maceral	Volume percent maceral composition					Average
	#1	#2	#3	#4	#5	
Huminite	64	68	64	64	64	65
Liptinite	6	5	5	4	8	6
Semifusinite	9	7	8	10	8	8
Fusinite	5	4	3	2	5	4
Other inertinites	11	13	7	8	10	10
Mineral matter	5	3	13	10	5	7

\*Same polished coal pellet analyzed on the same day using the same microscope.



**Table 7.** Detailed petrographic composition of handpicked huminite from Vesta mine (Horseshoe Canyon Formation)

Maceral group	Maceral	Volume percent (mmfb)	
		Sample #1	Sample #2
Huminite	Humotelinite	Textinite	nil
		Texto-ulminite	5
		Eu-ulminite	80
	Humocollinite	Gelinite	nil
		Corpohuminite	14
	Humodetrinite	Attrinite	nil
		Dessinite	1
	Total		100
			87
Liptinite	Sporinite	nil	3
	Cutinite	nil	1
	Resinite	nil	2
	Alginite	nil	nil
	Liptodetrinite	nil	5
	Total	nil	11
Inertinite	Semifusinite	nil	1
	Fusinite	nil	2
Mineral matter			1
% Reflectance		0.42	0.45

**Figure 3.** Relationship between H/C atomic ratios and (a) combined huminite-liptinite contents and (b) combined semifusinite-fusinite contents

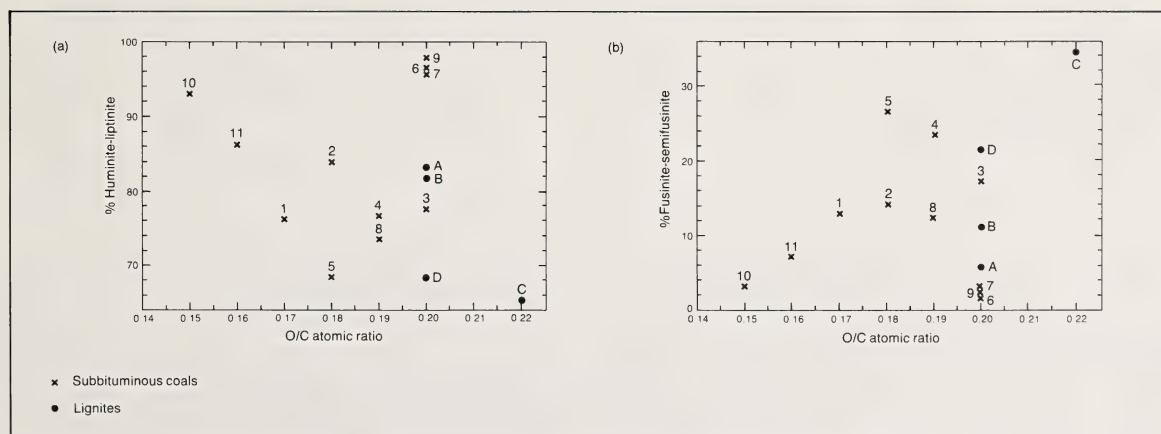
changes upon oxidation, a coal sample from the Blackfoot region (Horseshoe Canyon Formation) was oxidized in an oxygen plasma low temperature ashing unit at 150°C for different intervals of time. Compared

to a fresh sample, the petrographic composition of the oxidized portions showed only slight deformation for huminitic and liptinitic macerals (table 13). Under the experimental oxidation conditions, huminite was par-

**Table 8.** Elemental composition of subbituminous and lignite coals

Coal Identification	ASTM rank	Proximate analysis				Ultimate analysis (daf)					Calorific value of daf matter kJ/g
		Capacity moisture %	Ash (dry) %	VM (daf) %	Fixed carbon (daf) %	C %	H %	N %	S %	O % (by diff)	
Paskapoo Formation											
Smoky Tower	Sb A	16.0	20.0	43.4	56.6	75.8	5.1	1.7	0.5	16.9	29.8
Fox Creek	Sb B	17.6	17.0	40.0	60.0	75.8	4.2	1.1	0.5	18.4	29.7
Judy Creek	Sb C	nd	16.0	40.5	59.5	75.1	4.1	0.9	0.4	19.5	28.4
Highvale	Sb B	19.6	10.7	39.6	60.4	75.0	4.4	1.1	0.2	19.3	28.8
Ardley	Sb B	19.3	20.0	39.3	60.7	75.7	4.1	1.2	0.5	18.5	29.2
Horseshoe Canyon Formation											
Egg Lake	Sb C	25.2	14.6	43.2	56.8	74.2	4.5	1.6	0.4	19.3	28.3
Tofield	Sb C	23.3	24.4	43.8	56.2	73.4	4.4	1.6	1.2	19.2	27.9
Vesta	Sb C	24.6	12.2	41.9	58.1	74.4	5.0	1.5	0.6	18.5	29.4
Sheerness	Sb C	25.6	10.9	43.2	56.8	73.7	4.6	1.7	0.8	19.2	28.1
Blackfoot	Sb B	16.2	19.4	43.0	57.0	76.8	5.1	1.7	0.8	15.6	28.7
Oldman Formation											
Bow City	Sb B	15.7	13.7	47.1	52.9	75.8	5.4	1.8	0.9	16.1	30.6
Lignite											
Klimax, Sask.		8.8	17.0	43.2	56.8	73.3	4.5	1.2	1.2	19.8	17.9
Bienfait, Sask.		6.3	7.1	45.2	54.8	73.2	4.6	1.2	1.1	19.9	18.4
North Dakota		29.1	16.4	50.1	49.7	70.8	4.6	1.2	2.8	20.6	15.6
Texas		5.2	11.9	51.3	48.7	72.7	5.2	1.5	1.3	19.3	18.6

daf = dry, ash-free.

**Figure 4.** Relationship between O/C atomic ratios and (a) combined huminite-lipinite contents and (b) combined semifusinite-fusinite contents

tially devolatilized from edges which was reflected in the maceral composition. There were, however, no oxidation rims surrounding huminite, as are usually reported for bituminous coals. Nor did the reflectance of huminite show any appreciable change. This fact is also evident from the work of Chandra (1958), who studied the reflectance of coals oxidized in the

laboratory under controlled conditions at temperatures up to about 150°C. There are reports in the literature that the petrological compositions of samples of coal exposed to the atmosphere for a few years do not show any significant change (Chandra, 1966). From this study it is also evident that, as far as petrological analyses are concerned, storage of coal samples



**Table 9.** Petrographic composition variations in Highvale coal deposit

Sample location		Percent ash (dry)	Reflectance (percent)	Volume percent (mmfb)	
				Huminite plus liptinite	Inertinites
Top	#1	22.4	0.442	75	25
Seam #1	#2	8.4	0.408	79	21
	Partition				
	#3	14.2	0.372	83	17
Seam #2	#4	10.1	0.404	92	8
	#5	7.0	0.426	92	8
	#6	8.1	0.395	93	7
	#7	19.8	0.420	85	15
Bottom	#1 and #2 blended	13.4	0.446	75	25

**Table 10.** Petrographic composition variations in the Ardley (Paskapoo Formation) upper seam

Sample description	Ardley core hole #1-82			Ardley core hole #2-82			
	34	35	36	143	144	145	146
Distance from top (feet)							
Maceral composition (volume percent)							
Huminite	80.3	55.4	50.0	58.3	56.3	67.0	64.7
Liptinite	1.7	3.3	13.7	3.3	5.7	5.7	5.3
Semifusinite	5.7	12.7	12.7	9.7	14.7	11.7	9.3
Fusinite	2.0	16.3	11.0	17.7	10.3	10.0	6.7
Other inertinites	7.0	5.3	3.6	4.7	4.7	2.0	2.0
Mineral matter	3.3	7.0	9.0	6.3	8.3	3.6	12.0
Reflectance (%)	0.43	0.41	0.40	0.48	0.45	0.45	0.46

**Table 11.** Variations in petrographic composition\* of Vesta (Battle River) coal seam (Horseshoe Canyon Formation)

	Seam interval (ft) measured from top				
	0 to 0.5	0.9 to 1.4	1.9 to 2.4	2.9 to 3.1	4.4 to 4.9
Maceral	Percent volume maceral composition (mmfb)				
Huminite	92.8	93.3	89.6	96.4	94.3
Liptinite	1.4	1.8	2.3	2.0	2.4
Semifusinite	2.1	1.3	3.4	0.4	1.2
Fusinite	2.0	1.0	2.5	nil	1.2
Other inertinites	1.7	2.6	2.2	1.2	0.9

\*Average of two analyses.

**Table 12.** Petrographic composition of coal from different core holes in the Ardley region (W-4 meridian)\*

Maceral	CH-1	CH-2	CH-3	CH-4	CH-5
	SE Sec. 20 Tp 38, Ra 23	SW Sec. 6 Tp 39, Ra 24	SE Sec. 13 Tp 40, Ra 26	SE Sec. 29 Tp 41, Ra 24	NE Sec. 36 Tp 40, Ra 25
Volume percent					
Huminite	80.3	58.3	67.0	70.0	79.0
Liptinite	1.7	3.3	2.7	5.3	2.7
Semifusinite	5.7	9.7	11.0	6.0	6.3
Fusinite	2.0	17.7	6.3	5.0	4.0
Other inertinites	7.0	4.7	6.0	5.0	2.3
Mineral matter	3.3	6.3	7.0	8.7	5.7
Reflectance (percent)	0.43	0.48	0.45	0.42	0.47

\*From Alberta Geological Survey.

Tp = Township.

Ra = Range.

**Table 13.** Petrographic composition of Blackfoot coal, oxidized in oxygen plasma at low temperature (150°C) for different intervals of time

Maceral	Oxidation time (hrs)						
	0	1	2	3	4	5	6
Volume percent maceral composition							
Huminite	71	63	66	66	58	53	54
Liptinite	4	4	3	3	2	2	2
Semifusinite	1	nil	nil	nil	nil	1	nil
Fusinite	1	nil	nil	nil	nil	1	1
Other inertinites	2	nil	nil	1	nil	1	1
Mineral matter	21	33	31	30	40	45	44
Huminite reflectance (maximum) %	0.52	0.59	0.58	0.58	0.57	0.56	0.59

under ambient conditions is satisfactory, but storage in inert atmosphere or at least at low temperature is desirable for chemical analysis of technological tests.

### Reflectance determination

Reflectance for a given coal is reported as the mean of the individual readings of maximum or random reflectance. Rank determination is performed by measuring either maximum reflectance in polarized, reflected light, or random reflectance, usually in nonpolarized, reflected light. The choice between the two techniques depends upon individuals. Measuring maximum reflectance requires that the object stage be rotated until a maximum value of reflectance is recorded. For most purposes, the reflectance of the huminite (vitrinite) maceral of coal is measured. The reproducibility of the mean is of the order of  $\pm 0.02$  percent reflectance at the 95 percent confidence level

for representative samples of a coal. Typical random reflectance output data from the computer for a subbituminous coal from the Paskapoo Formation (Ardley) is shown in table 14. In evaluating a coal for a technical process, reflectance distribution is a very practical approach.

Reflectance data on huminite for the subject coals are included in table 4. Figure 5 shows the random reflectance distribution for the subbituminous coals. For the subbituminous coals, the reflectance can be compared with the rank classification determined by ASTM procedures. Another way of describing reflectance data is to compute percentages of readings at various reflectance levels (so-called 1/2-V type<sup>1</sup>).

<sup>1</sup>Each 1/2-V type represents a reflectance range of 0.05 percent; for example, all readings in an analysis from 0.40 to 0.44 percent are assigned the midpoint to 1/2-V type 4.24.



Reflectance as a 1/2-V type distribution for the tested coals is given in table 15 and plotted in figure 6 for three coals. For the subbituminous coals, the reflectance can be compared with rank classification determined by ASTM procedures. The highest reflectance obtained on the subbituminous A coal (Smoky Tower) is 0.47. Average reflectances for the subbituminous B and C coals are respectively 0.45 and 0.41.

Reflectance values for the lignites (table 4) are lower than those obtained on the subbituminous coals and indicate the following order of coalification: Saskatchewan lignite — Texas lignite — North Dakota lignite.

In coals below the rank of low volatile bituminous, huminite (vitrinite) always behaves as a uniaxial negative substance. Consequently, a surface that is perpendicular to the bedding displays a maximum refractive, and thus reflectance, index when the inci-

dent light is polarized in a direction parallel to the bedding, and a minimum reflectance when the light is polarized perpendicular to the bedding; the difference between the maximum and minimum reflectances is termed bireflectance. On an oblique coal surface, maximum reflectance is displayed parallel to the bedding; intermediate values between maximum and minimum are shown at right angles to the direction. A maximum reflectance value can, therefore, always be obtained with a reflectometer by rotating any coal particle under microscope until the bedding direction is parallel to the plane of vibration of the polarized incident light. The anisotropic character of huminite (vitrinite) is shown in figure 7 (Davis 1978).

Maximum and minimum reflectances of the huminite of four subbituminous coals (Smoky Tower, Ardley, Highvale and Vesta) were measured and an index of

**Table 14.** Reflectance distribution measurements: huminite random reflectance for Ardley coal

0.432	0.452	0.443	0.481	0.478	0.455	0.465	0.472	0.469	0.442
0.458	0.421	0.437	0.441	0.413	0.448	0.453	0.471	0.415	0.482
0.448	0.459	0.469	0.463	0.385	0.466	0.448	0.464	0.478	0.457
0.460	0.443	0.466	0.480	0.478	0.459	0.443	0.448	0.475	0.453
0.469	0.473	0.427	0.449	0.465	0.414	0.455	0.404	0.473	0.443
Arithmetic mean					N = 50				
Standard deviation					X5 = 0.45				
Variance					SIGXI = 0.02				
Standard deviation of mean					VARX = 0.00				
Standard error of standard deviation					SIGXB = 0.00				
Confidence level					3(SIGXI) = 0.00				
					L95 = 0.01				
					L99 = 0.01				

**Table 15.** 1/2-V type reflectance distribution in subbituminous coals

Coal	½V-type									Average random reflectance (percent)
	2.25	2.75	3.25	3.75	4.25	4.75	5.25	5.75	6.25	
	Percent of total counts									
Smoky River	0	0	0	12	25	41	18	4	0	0.465 ± 0.051
Fox Creek	0	0	0	28	46	26	0	0	0	0.427 ± 0.035
Judy Creek	0	0	10	72	16	2	0	0	0	0.379 ± 0.026
Highvale	0	0	8	18	51	8	8	7	0	0.414 ± 0.083
Ardley	0	0	2	6	44	38	4	4	2	0.452 ± 0.048
Egg Lake	0	0	12	22	28	36	2	0	0	0.422 ± 0.054
Tofield	0	0	12	24	32	16	16	0	0	0.422 ± 0.062
Vesta	2	6	40	40	8	4	0	0	0	0.358 ± 0.044
Castor	0	0	0	4	32	40	16	8	0	0.474 ± 0.049
Sheerness	0	0	2	6	20	44	26	2	0	0.472 ± 0.042
Blackfoot	0	0	0	12	32	24	24	8	0	0.474 ± 0.060
Bow City	0	0	0	8	56	24	10	2	0	0.445 ± 0.039
Rolling Hills	0	0	0	8	42	46	2	2	0	0.450 ± 0.035

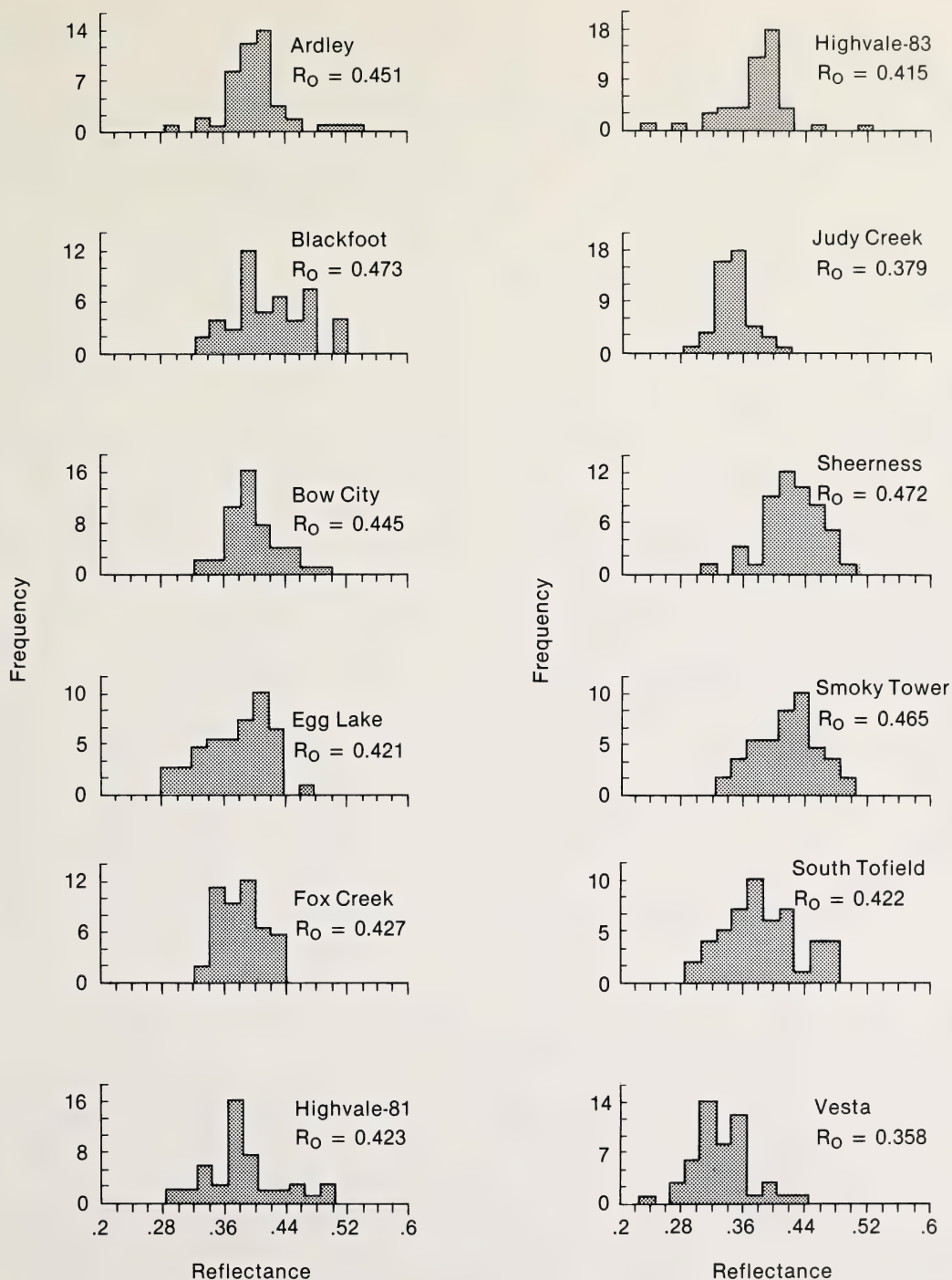
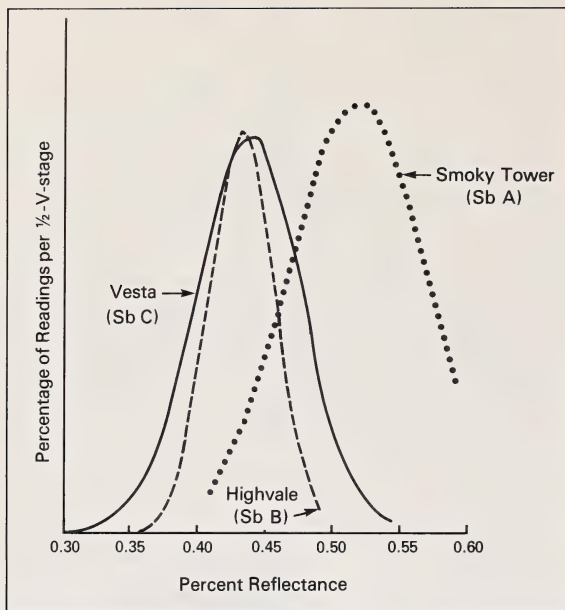
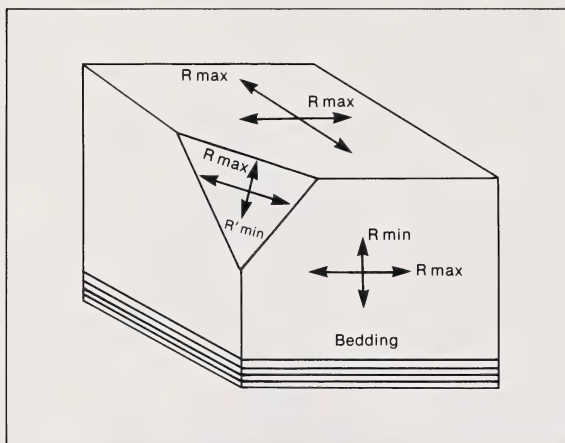


Figure 5. Reflectance distribution in Alberta plains coals





**Figure 6.** Reflectance frequency distribution diagram for subbituminous coals



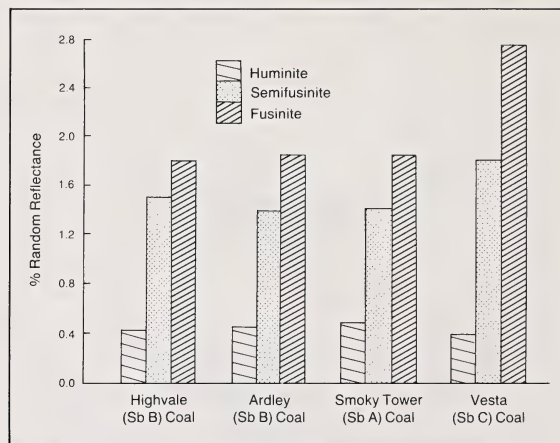
**Figure 7.** The anisotropic character of vitrinite

the anisotropy of the huminite of these four coals was calculated (table 16). These data should be useful in accounting for the amount of coke formation during any thermal treatment (pyrolysis or liquefaction).

Few data have been published on the reflectances of macerals other than huminite (vitrinite). To obtain a better picture of the degree of metamorphism of Alberta subbituminous coals, reflectance of semifusinite and fusinite was measured. A comparison between the mean reflectance for different macerals is summarized in table 17, which shows that fusinite is the highest reflectance maceral (figure 8).

The reflectance distribution of different macerals, shown in figure 9, for a coal from Oldman and Foremost Formations, indicates the degree of petrographic heterogeneity in subbituminous coals. Extensive overlapping of macerals in terms of reflectance distribution shows that a fair amount of low reflectance semifusinite is closely associated with huminite, which is perhaps fairly reactive in a technical process.

A variety of automated reflectance measurement techniques have been developed in recent years which are ideal for quantitative characterization of a coal. This eliminates the need for tedious quality control analysis procedures otherwise required. A sample analysis for a subbituminous coal (Vesta) using the Im-



**Figure 8.** Reflectance of subbituminous coal macerals

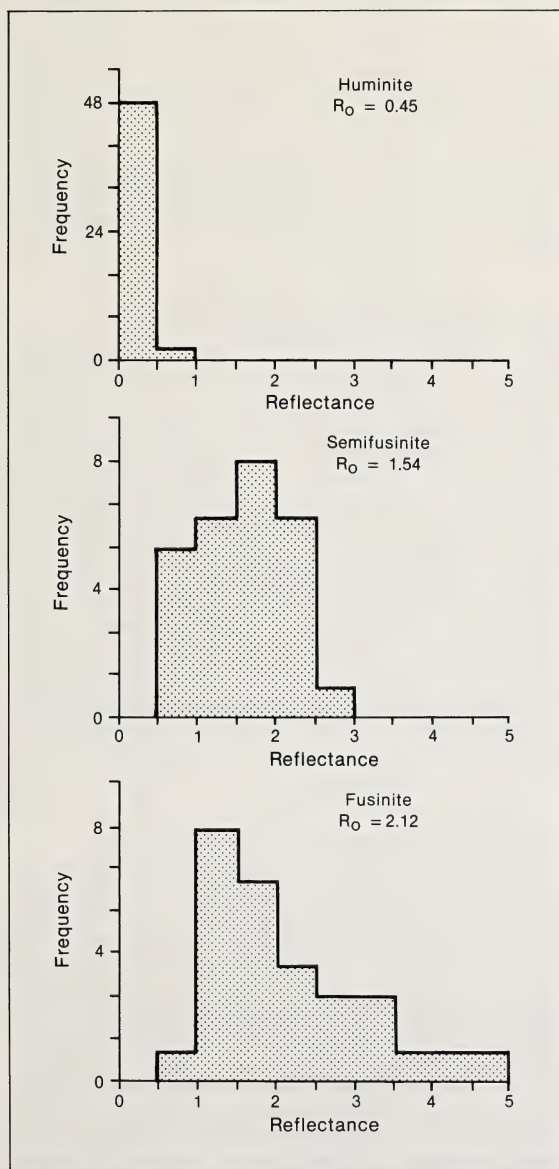
**Table 16.** Anisotropy of huminite in subbituminous coals

Coal	Percent reflectance		Bireflectance ( $R_{\max} - R_{\min}$ )	Anisotropy index ( $R_{\max}$ ) ( $R_{\max} - R_{\min}$ )
	$R_{\max}$	$R_{\min}$		
Smoky Tower	0.51	0.46	0.05	10.2
Ardley	0.48	0.33	0.15	3.2
Highvale	0.46	0.36	0.10	4.6
Vesta	0.46	0.36	0.10	4.6

$R_{\max}$  = Maximum reflectance.  $R_{\min}$  = Minimum reflectance.

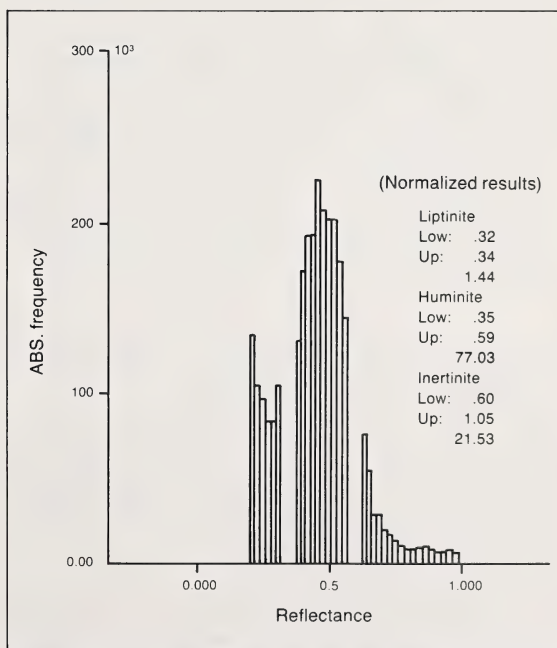
**Table 17.** Random reflectance of huminite, semifusinite and fusinite macerals in subbituminous coals

Coal	ASTM rank	Percent random reflectance of:		
		Huminite	Semifusinite	Fusinite
Smoky Tower	Sb A	0.47	1.42	1.86
Ardley	Sb B	0.45	1.40	1.82
Highvale	Sb B	0.41	1.54	1.68
Vesta	Sb C	0.36	1.83	2.96

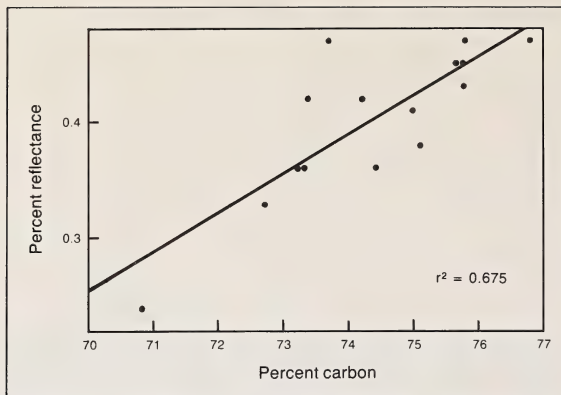
**Figure 9.** Reflectograms for different macerals in a subbituminous coal

age Analyser IBAS (Zeiss) available at the Institute of Sedimentary and Petroleum Geology, Calgary, is shown in figure 10. The results are quite comparable to those obtained by the point count method.

Reflectance data have long been recognized as reliable indicators of maturation for coals of bituminous and higher rank (McCartney and Teichmüller, 1972); excellent correlations have been established for volatile matter and carbon content. The reliability of reflectance for coals lower in rank than bituminous has been doubtful, although Marchioni (1979) showed good relationships in his study of the lignite-subbituminous coals of Hat Creek, British Columbia. The data reported in the present study show a good relationship of reflectance to carbon content (figure 11). A linear correlation between reflectance and carbon content for low rank coals (70-80 percent dry, ash-free carbon) has also been reported by van Krevelen (1981), Davis (1965) and McCartney and Teichmüller (1972).

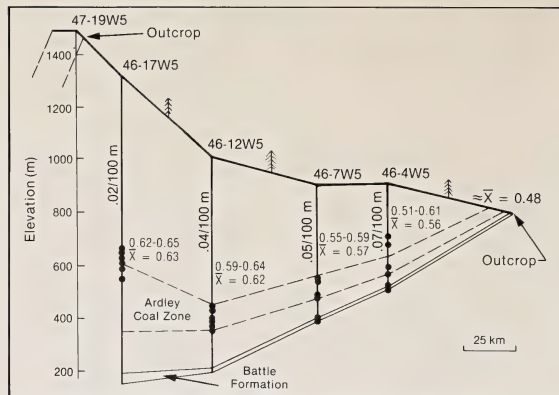
**Figure 10.** Reflectogram of Vesta coal using image analyzer





**Figure 11.** Relationship between random reflectance of huminite and carbon content

Figure 12 shows variations in rank for 22 coal samples covering a cross section from the Coal Valley area (in township 47, range 19W5) to the Ardley coal outcrop (in township 46, range 26W4) reported by



**Figure 12.** Quality variation in Ardley coals (Reflectance range of vitrinite)

Nurkowski (1984). The reflectance (maximum,  $R_M$ ) ranges from 0.51 to 0.65 and the reflectance gradient ( $R_M/100$  meter) varies from 0.07 to 0.02 from east to west.

## Concentration and separation of coal macerals

As coal science progresses, it becomes obvious that to evaluate the precise role of different organic coal components in technical processes, pure macerals are needed. The chemical and physical properties of various macerals are known to differ substantially (van Krevelen, 1981). Depending on their quantitative participation and their association, macerals control the chemical, physical and technological properties of a coal of a given rank (ICCP, 1983). Huminite and fusinite are the only coal macerals that can be macroscopically selected in an almost pure state. Lipinites and micrinite are invariably mixed with other macerals; their properties can only be derived from the behavior of concentrated fractions. Little attention has been given to the actual procedures for separating the macerals. In the present investigations, two methods were employed to obtain maceral concentrates from subbituminous coals.

### Preparation of maceral concentrates: float-and-sink technique

The true density of lipinites is lower and that of inertinites (semifusinite and fusinite) is higher than that of huminite. It is these differences that underly the technique of fractionating the macerals by means of liquids differing in specific gravity: the float-and-sink method.

#### Procedure of fractionation

The float-and-sink technique, using mixtures of halogenated and nonhalogenated hydrocarbons (cyclohexane, tetrachloroethylene, carbon tetrachloride and bromoform) of different densities (in the range 1.35 to 1.60  $\text{gcm}^{-3}$ ) was employed to obtain

different density fractions of subbituminous and lignite coals.

Approximately 100 grams of a coal (oven-dried, minus 100 mesh) were immersed in one litre of organic solvent with a density of 1.35  $\text{gcm}^{-3}$  at room temperature. The mixture was stirred for a few hours, using a magnetic stirrer, and then centrifuged. The float and sink portions were removed separately and dried thoroughly in a vacuum oven (1-2 mm Hg) at 40-45°C for 24 hours. The sink material was re-immersed in a fresh solution of a higher density and the procedure was repeated. After the fractions thus obtained were steamed and dried under vacuum, their densities, chemical and petrographic compositions were determined. Figure 13 gives a schematic procedure for obtaining density fraction of subbituminous coal.

#### Chemical and petrographic compositions

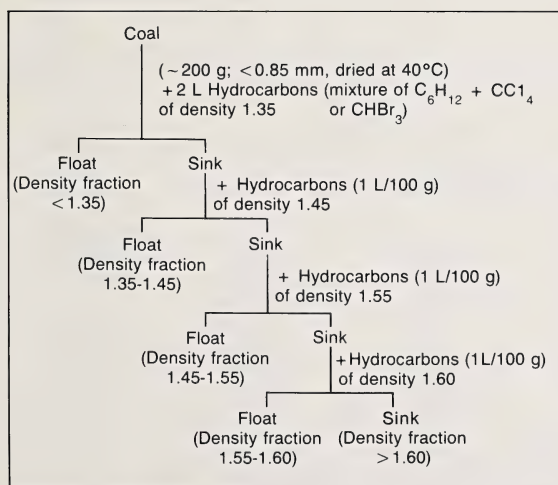
Tables 18 and 19 give the weight percent of density fractions of two subbituminous coals (Highvale-Paskapoo Formation and Vesta-Horseshoe Canyon Formation) and the two American lignites, as well as the true densities of the fractions (defined as the weight of a unit volume of the pore-free solid) measured by the helium displacement method. In Vesta coal, approximately 44 percent of the sample belongs to density fraction 1.45 to 1.55; for Highvale coal this fraction constitutes only 26 percent. In Texas lignite, 73 percent of the sample belongs to density fraction 1.35 to 1.45; in North Dakota lignite this fraction constitutes only 40 percent. The true densities measured experimentally for all the fractions fall within the density range used for their separation, which in-

**Table 18.** Percentage density fractions of subbituminous coals

Fraction density (gcm <sup>-3</sup> )	Highvale coal subbituminous B (Paskapoo Formation)		Vesta coal subbituminous C (Horseshoe Canyon Formation)	
	True density in helium (gcm <sup>-3</sup> )	Weight percent (dry basis)	True density in helium (gcm <sup>-3</sup> )	Weight percent (dry basis)
< 1.35	1.33	13.8	1.34	20.6
1.35-1.45	1.41	39.3	1.40	20.7
1.45-1.55	1.49	25.8	1.52	43.7
1.55-1.60	1.57	7.8	1.58	4.0
> 1.60	1.66	13.3	1.65	11.0

**Table 19.** Percentage density fractions of lignites

Fraction density (gcm <sup>-3</sup> )	Texas lignite		North Dakota lignite	
	True density in helium (gcm <sup>-3</sup> )	Weight percent (dry basis)	True density in helium (gcm <sup>-3</sup> )	Weight percent (dry basis)
< 1.35	1.34	15.2	1.36	0.6
1.35-1.45	1.43	73.4	1.42	40.3
1.45-1.55	1.51	5.7	1.46	33.7
1.55-1.60	1.57	0.7	1.58	7.8
> 1.60	1.65	5.0	1.65	17.6

**Figure 13.** Schematic procedure for separation of density fractions of coal

indicates that the fractionation procedure is quite satisfactory.

The chemical analyses of the density fractions of subbituminous coals (Highvale subbituminous B and Vesta subbituminous C) and the two American lignites are given in tables 20 and 21 respectively. As ex-

pected, the ash content increases progressively with an increase in fraction densities and various fractions differ considerably in their chemical compositions.

Tables 22-25 give the petrographic composition and average random reflectance for the density fractions of two subbituminous coals (Highvale subbituminous B and Vesta subbituminous C) and two American lignites. It is apparent from the petrographic analyses that:

1. For density fraction <1.35 and 1.35 to 1.45, the combined huminite and liptinite contents are higher than the respective values for higher density fractions.
2. There is no significant difference in the reflectance of different density fractions for either of the subbituminous coals or lignites studied. This seems to result from the fact that the mean reflectance of a huminite in a given coal is largely uninfluenced by variation in petrographic composition, although liptinite content may influence reflectance in some coals (Kalkreuth, 1982).

The photomicrographs (plates 1 to 4) give a general idea of how different macerals are distributed in different density fractions of subbituminous and lignite coals.

In figure 14 the H/C ratios for the density fractions are plotted against their combined huminite-liptinite contents. At first glance, the points seem to be scattered completely at random. A closer inspection of the



**Table 20.** Chemical analysis of density fractions of subbituminous coals

Fraction density (gcm <sup>-3</sup> )	Highvale coal (subbituminous B)					Vesta coal (subbituminous C)				
	Dry basis percent					Dry basis percent				
	Ash	C	H	N	(O+S+ Halogens)	Ash	C	H	N	(O+S+ Halogens)
Composite	10.7	67.0	3.9	1.0	17.4	12.2	65.3	4.4	1.3	16.8
< 1.35	4.0	71.7	4.4	0.8	19.1	5.3	69.6	4.3	1.6	19.2
1.35-1.45	5.3	70.8	4.2	0.9	18.8	6.3	69.2	4.3	1.6	18.6
1.45-1.55	8.5	68.8	3.7	1.0	18.0	10.3	65.9	4.2	1.6	18.0
1.55-1.60	11.7	66.4	3.4	0.9	17.6	17.5	60.4	3.9	1.7	16.5
> 1.60	42.9	39.1	2.5	0.7	14.8	52.5	33.9	2.5	0.9	10.2

**Table 21.** Chemical analysis of density fractions of U.S. lignites

Fraction density (gcm <sup>-3</sup> )	North Dakota lignite					Texas lignite				
	Dry basis percent					Dry basis percent				
	Ash	C	H	N	(O+S+ Halogens)	Ash	C	H	N	(O+S+ Halogens)
Composite	17.1	58.7	3.8	1.0	19.4	12.8	63.4	4.5	1.3	18.0
< 1.35	4.7	69.4	5.0	0.8	20.1	4.7	69.8	5.1	0.8	19.6
1.35-1.45	8.3	65.0	4.6	1.0	21.1	6.9	63.2	4.6	1.2	24.1
1.45-1.55	11.2	61.8	3.9	1.1	22.0	9.5	59.9	4.2	1.2	25.2
1.55-1.60	13.8	60.7	3.9	1.1	20.5	15.0	56.3	4.1	1.3	23.3
> 1.60	28.9	47.5	2.9	0.9	19.8	29.5	47.4	3.6	1.0	18.5

**Table 22.** Maceral analysis of density fraction of Highvale coal (subbituminous B)

Fraction density (gcm <sup>-3</sup> )	Volume percent maceral composition (mmf)					Average random reflectance of huminite (percent)
	Huminite	Liptinite	Semi-fusinite	Fusinite	Other inerts	
Composite	74.6	2.1	22.2	1.1	—	0.48
< 1.35	95.9	0.7	2.8	nil	0.6	0.46
1.35-1.45	74.9	6.3	7.1	2.3	9.4	0.43
1.45-1.55	50.5	3.7	37.6	3.4	4.8	0.43
1.55-1.60	48.0	1.9	26.7	18.3	5.1	0.43
> 1.60	41.3	0.9	30.4	18.0	9.4	0.44

figure shows, however, that there may be at least two factors other than huminite-liptinite content affecting the distribution of the plotted points. One of these factors is rank, the other is mineral matter content. Population 1 (in figure 14) consists entirely of subbituminous samples with relatively low ash (mineral matter) contents; thus the major influence appears to be the huminite-liptinite content). This population is

concentrated in one part of the diagram. Population 2 consists of a mixture of subbituminous and lignite samples all characterized by high ash contents. Their H/C ratios appear to be too high for their huminite-liptinite content and they follow a different trend line from that shown by population 1. In population 2 the mineral matter content appears to be the significant factor. In between is a rather diffuse group of points

**Table 23.** Maceral analysis of density fractions of Vesta coal (subbituminous C)

Fraction density (gcm <sup>-3</sup> )	Volume percent maceral composition (mmf)					Average random reflectance of huminite (percent)
	Huminite	Liptinite	Semi-fusinite	Fusinite	Other inerts	
Composite	73.3	0.4	8.9	3.3	14.1	0.41
< 1.35	89.5	5.3	2.8	1.0	1.4	0.43
1.35-1.45	91.0	2.8	2.1	1.0	3.1	0.46
1.45-1.55	82.8	1.9	6.8	2.9	5.6	0.44
1.55-1.60	76.2	1.5	6.0	5.6	10.7	0.47
> 1.60	72.5	1.5	5.0	7.3	13.7	0.45

**Table 24.** Maceral analysis of density fraction of Texas lignite

Fraction density (gcm <sup>-3</sup> )	Volume percent maceral composition (mmf)					Average random reflectance of huminite (percent)
	Huminite	Liptinite	Semi-fusinite	Fusinite	Other inerts	
Composite	60.9	7.6	10.9	10.5	10.1	0.34
< 1.35	71.6	9.1	4.4	6.5	8.4	0.36
1.35-1.45	79.7	9.4	2.3	3.3	5.3	0.31
1.45-1.55	82.0	4.7	5.4	1.2	6.7	0.30
1.55-1.60	47.9	5.2	15.9	17.3	13.7	0.29
> 1.60	47.3	5.9	18.5	17.5	10.8	0.31

**Table 25.** Maceral analysis of density fraction of North Dakota lignite

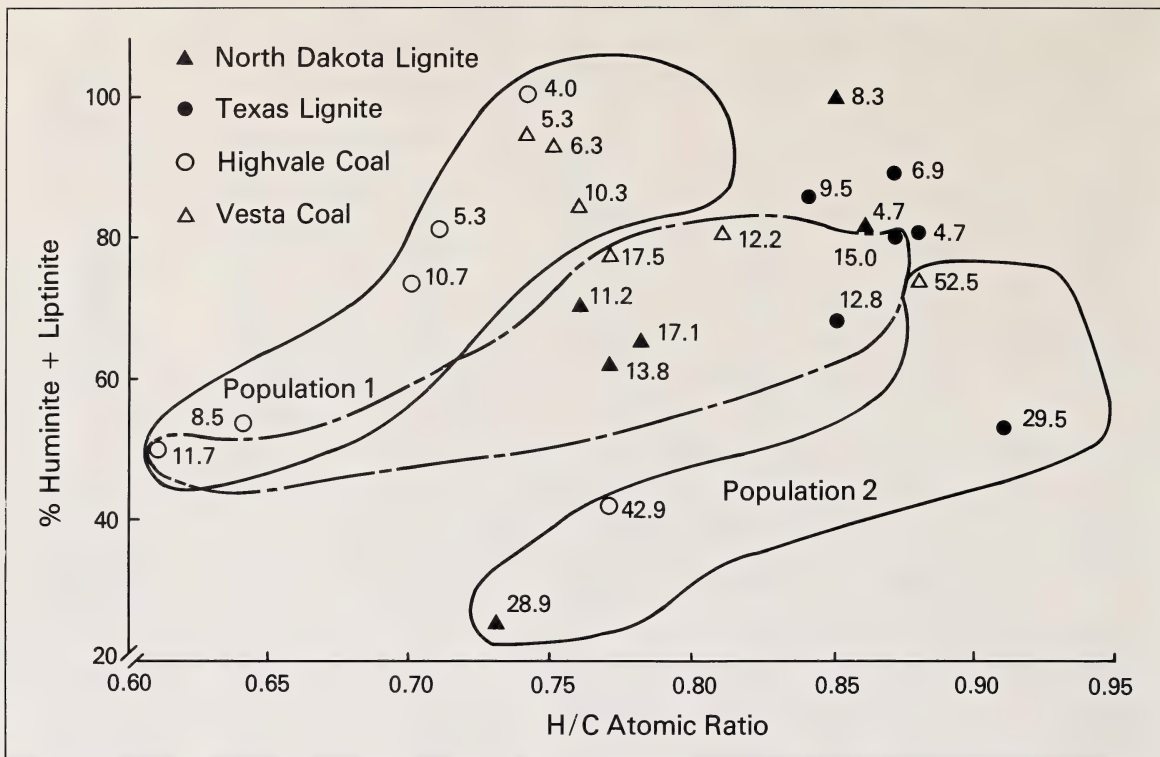
Fraction density (gcm <sup>-3</sup> )	Volume percent maceral composition (mmf)					Average random reflectance of huminite (percent)
	Huminite	Liptinite	Semi-fusinite	Fusinite	Other inerts	
Composite	60.3	5.3	22.1	12.3	nil	0.24
< 1.35	77.4	4.8	15.2	2.6	nil	0.21
1.35-1.45	89.5	6.4	4.1	nil	nil	0.26
1.45-1.55	66.9	3.1	25.8	4.2	nil	0.26
1.55-1.60	59.3	2.9	31.9	5.9	nil	0.28
> 1.60	22.6	2.9	40.3	34.2	nil	0.28

consisting mainly of lignite samples with low to intermediate ash contents. The main difference between these samples and those of population 1 appears to be rank. This can be demonstrated by comparing lignite samples with subbituminous samples of similar ash and huminite contents: in every case, the lignite samples shown the higher H/C ratio. Figures 3 and 14 suggest that, when plotting the elemental composition of coals, besides maceral compositions, rank and ash content should be taken into account.

## Separation of coal macerals: density gradient centrifugation technique

In order to demonstrate applications to subbituminous coals, the density gradient centrifugation technique (DGC) to isolate pure macerals from bituminous coals proposed at the Argonne National Laboratory (Dyrkacz and Hortwitz, 1982) was tried. Similar to the float-and-sink technique, the DGC method also takes advantage





**Figure 14.** Relationship between H/C atomic ratios and combined huminite-liptinite contents of coals and their density fractions

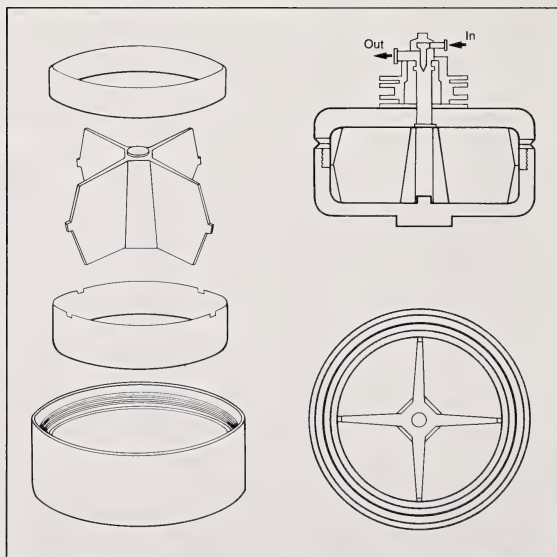
of the known density differences of the various macerals.

#### Maceral separation scheme

Three key elements necessary for the successful separation of macerals using the density gradient method are:

1. proper grinding to free macerals
2. demineralization
3. complete dispersion of fine coal particles

Preparative-scale separation of macerals was done using a zonal rotor which can be used with 1.4 L of density gradient liquid (figure 15). The density gradients were preformed using a commercial gradient maker (LKB 1300 Ultragrad Gradient Mixer). Aqueous cesium chloride was used to make up the desired densities (1.20-1.65 gcm<sup>-3</sup> range). Two stock solutions, representing the maximum and minimum densities of the gradient, were required to gradient formation. The gradient was loaded while the Zonal rotor was spinning at 2000 rpm. The coal suspension, consisting of about 2 g of demineralized coal (less than 30 microns average particle size) in 100 mL of water containing surfactant (Brij-35), was added after subjecting the slurry to intermittent cycle sonication. After loading, the system was accelerated to 12000 rpm and run at this speed for one hour. The rotor was then decelerated to 2000 rpm and the contents pumped out to a fraction collector using the floriant dense chase

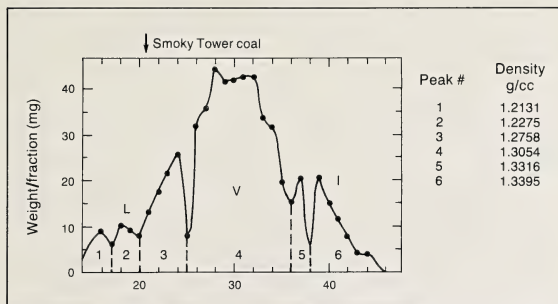


**Figure 15.** Zonal rotor used for maceral separations

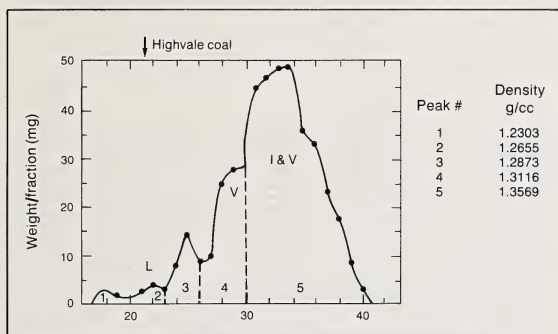
solution. The density of each fraction was measured. Each fraction was filtered using a 0.8 micron polycarbonate nuclepore membrane filter and washed successively, first with 30 to 50 mL of distilled water at

room temperature and at about 80°C, then with 95 percent ethanol. Each washed fraction was dried overnight at 40°C in a vacuum oven and weighed accurately.

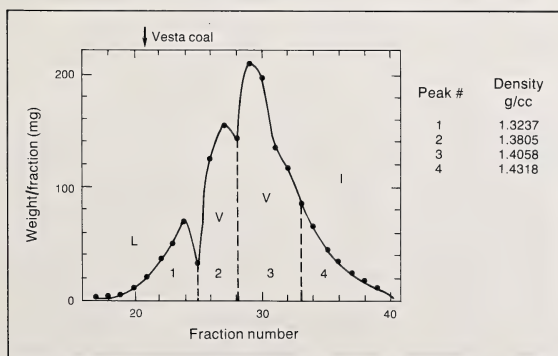
Typical DGC runs for the three chemically demineralized coals (Smoky Tower, Highvale and Vesta) are shown in figures 16-18. The weight data for each fraction separated from the three coals showed differences in the positions and shapes of the curves. The position of the peaks, even without microscopic identification, allowed a crude estimate of the amount of each maceral in each sample.



**Figure 16.** Analytical density gradient centrifugation of Smoky Tower (subbituminous A) coal



**Figure 17.** Analytical density gradient centrifugation of Highvale (subbituminous B) coal



**Figure 18.** Analytical density gradient centrifugation of Vesta (subbituminous C) coal

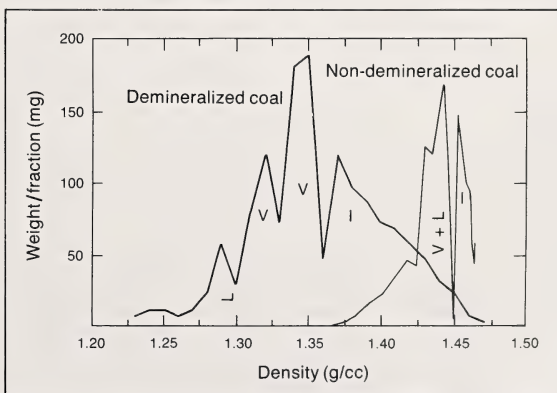
Petrographic analysis of DGC separation fractions is given in table 26. Resolutions of 90 percent (by volume) or greater purity were obtained in some fractions with a single DGC run. The low density inertinite (micrinite) in all the tested coals was problematic for better resolution. Resolution of macerals in Highvale coal was somewhat inferior to that of the other two coals, probably because of the presence of a significantly higher amount of semifusinite. The task of separating macerals from subbituminous coals appeared to be much more difficult than from bituminous coals.

In regard to the petrographic analysis of DGC separated fractions, it is appropriate to mention that with finely-ground coal the morphology was usually lost and the only identifying parameter for distinguishing a maceral was the reflectance measurement. To see particles adequately, magnifications greater than 1000 times were needed. Huminite and inertinite could be identified relatively easily but lipinites were often not clearly defined.

In the DGC maceral separation technique, the use of demineralized coals is a key element for successful runs. In the investigations of maceral reactivity, however, demineralization is not desirable. Figure 19 shows two analytical DGC runs using Vesta coal before and after demineralization with acid treatment. With no demineralization (12 percent mineral matter) the separation of macerals was quite diffused and generally the peaks for different macerals overlapped.

In an attempt to improve upon maceral separation procedures, 12 DGC runs were made on maceral concentrates obtained by float-and-sink technique. The results recorded in figures 20 and 21 indicated that demineralized coal, of course, resulted in better maceral separation but if the density fractionation of the subject coal was done previously using the float-and-sink method, it was possible to have far better resolution of different macerals.

One of the major limitations of the DGC maceral separation procedure was the small amount of material that was obtained in a single DGC run. The experience gained after several repeated experiments



**Figure 19.** Maceral separation using density gradient centrifugation method - effect of mineral matter removal



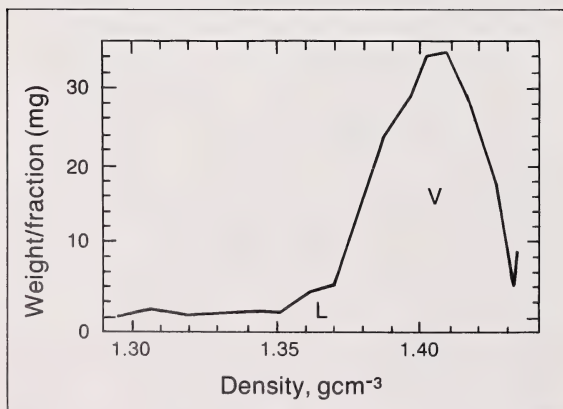
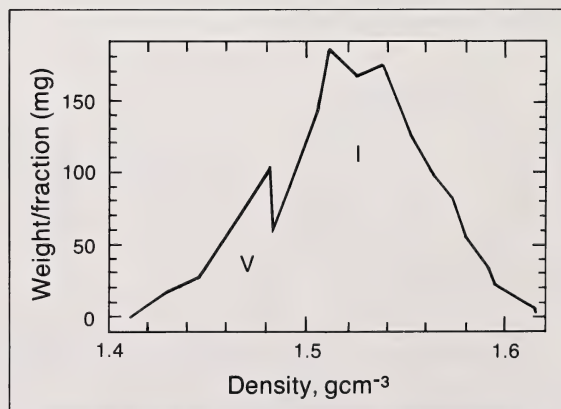
**Table 26.** Petrographic composition of isolated macerals from Vesta subbituminous coal using density gradient centrifugation method

Maceral		Fraction 1 density 1.23-1.32 gcm <sup>-3</sup>	Fraction 2 density 1.32-1.38 gcm <sup>-3</sup>	Fraction 3 density 1.38-1.41 gcm <sup>-3</sup>
Volume percent				
Liptinite	Sporonite	20	—	—
	Resinite	5	—	—
	Cutinite	39	—	—
	Alginite	1	—	—
	Total liptinite	64	—	—
Huminite	Textinite	—	—	—
	Eu-ulminite	21	64	14
	Humodetrinite	3	9	2
	Humocollinite	6	19	6
	Total huminite	30	92	22
Inertinite	Semifusinite	2	3	32
	Fusinite	2	2	38
	Micrinite	—	1	2
	inertodetrinite	1	2	16
	Total inertinite	5	8	78

for maceral separation from subbituminous coals suggested that multi-stage float-and-sink tests followed by the density gradient centrifugation could be a practical approach for obtaining pure macerals for tests in micro-reactors.

A procedure that was standardized for the separation of the maceral constituents of subbituminous coals involved two steps: concentration of macerals using the float-and-sink method; and isolation of pure

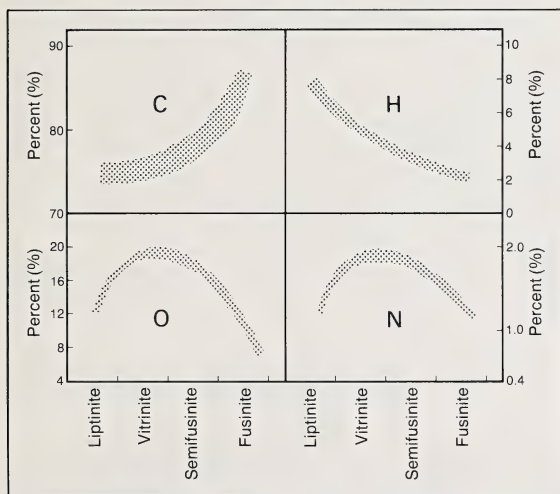
macerals from maceral concentrate fractions using the density gradient centrifugation technique. To isolate macerals in significant quantities, however, several runs were required which was time consuming indeed. For maceral reactivity studies (liquefaction, pyrolysis or combustion), where somewhat larger quantities of macerals were needed, a compromise between quality and quantity was the solution unless handpicked macerals collected at the mine site were acceptable.

**Figure 20.** Maceral separation using density gradient centrifugation method from low density fraction (< 1.45 gcm<sup>-3</sup>) of Highvale coal**Figure 21.** Maceral separation using density gradient centrifugation method from 1.45-1.60 gcm<sup>-3</sup> density fraction of Highvale coal

## Chemical composition of individual subbituminous coal macerals

Following Dormans *et al.* thesis (1957), the chemical composition of pure macerals from the petrographic analyses of subbituminous coal maceral concentrates has been calculated using balance equations. The results are recorded in table 27 and plotted in figure 22. The huminite macerals are relatively rich in oxygen, whereas the liptinite macerals are relatively rich in hydrogen. On the other hand, the inertinite macerals are relatively rich in carbon or, more in keeping with the other two maceral groups, are actually hydrogen-deficient. The average densities of macerals of the subbituminous coals, determined by the helium displacement method, are 1.285, 1.345 and 1.458 gcm<sup>-3</sup> (dry, ash-free basis) for liptinite, huminite and inertinite respectively. On the basis of these significant differences in composition, it is expected that the three maceral groups will show different behavior in many of the technological processes for coal utilization.

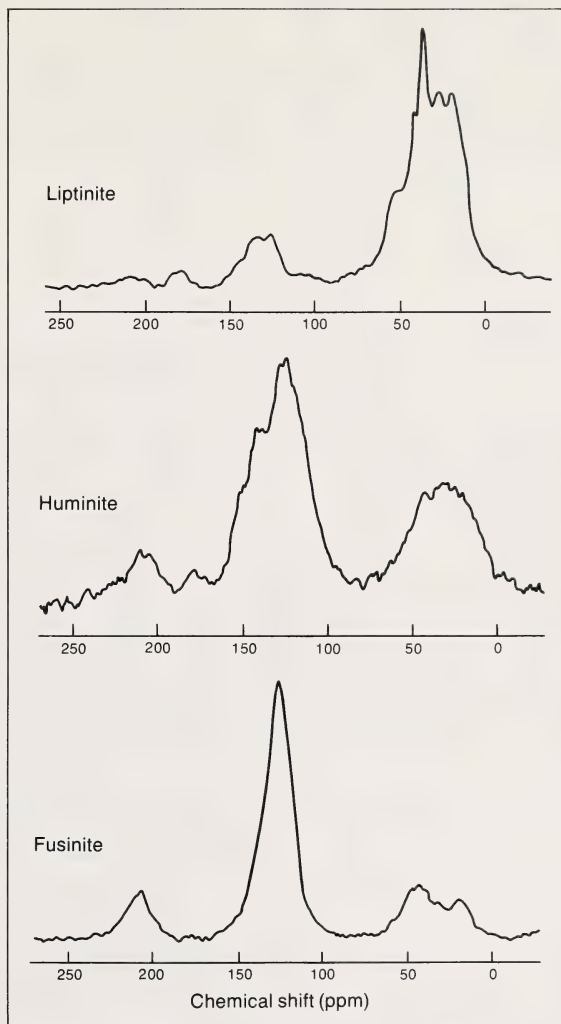
X-ray diffraction studies of subbituminous coal macerals showed a very low degree of ordering of the atoms. In very restricted areas, however, the atoms were better ordered and thus only rough estimates of the ultrafine structure of molecules could be made by x-ray examination. The interplanar crystalline spacings increased in the order: fusinite — vitrinite — liptinite. In fusinite, aromatic lamellae (about 0.21 nm in diameter)



**Figure 22.** Chemical composition of subbituminous coal maceral

were parallel at a distance of about 0.32 nm and formed stacks less than 0.1 nm in height; aromatic carbon occurred in layers composed of less than three condensed rings.

The CP/MAS C-13 NMR (cross-polarization/magic angle sample spinning nuclear magnetic resonance) spectra of three macerals (liptinite, huminite and inertinite) were obtained (figure 23) and the wide diversity



**Figure 23.** CP/MAS spectra of subbituminous coal macerals

**Table 27.** Chemical composition of subbituminous coal macerals (percent)

	Liptinite	Huminite	Semifusinite	Fusinite
Carbon	73.8	74.6	76.5	88.6
Hydrogen	8.1	4.9	3.6	2.5
Oxygen	16.4	18.2	17.7	7.6
Nitrogen	1.2	1.8	1.7	1.1
Sulfur	0.5	0.5	0.5	0.2



in chemical structure of these species will be the subject of a future detailed report. The liptinite maceral, for instance, exhibited an unusually high degree of resolution among the various aliphatic carbon functionalities

that had enabled a much less ambiguous analysis of relaxation behavior than previously reported (Zilm *et al.*, 1981; Maciel *et al.*, 1982; Wilson *et al.*, 1984).

## Petrography with reference to liquefaction potential

### Liquefaction behavior of maceral concentrates

To investigate the liquefaction behavior of maceral concentrates, two liquefaction techniques were employed: the I.G. Farben type direct hydroliquefaction process and the CO/steam type process. The experimental conditions were as follows:

	Farben type process	CO/Steam type process
Sample size	10 g dry, ash-free coal	10 g dry, ash-free coal
Solvent	20 g anthracene oil	20 g anthracene oil
Water	--	1 mole/mole CO
Catalyst	Fe <sub>2</sub> O <sub>3</sub> (3 percent by weight of coal)	K <sub>2</sub> CO <sub>3</sub> (5 percent by weight of coal)
Initial pressure	6.0 MPa (H <sub>2</sub> )	5.5 MPa (CO)
Reaction temperature	460°C	400°C
Reaction time	10 minutes	10 minutes

The appropriate quantities of coal, catalyst, solvent and water (for the CO/steam process) were charged into a tubing bomb mini-autoclave (130 mL effective volume). The tube bomb was purged and subsequently pressurized with hydrogen (6.9 MPa) or carbon monoxide (5.5 MPa) and immersed into the fluidized sand bath, which was heated to the desired liquefaction temperature. During the experiment, the tube bomb was continuously agitated vertically using a motor-pulley system. Immediately after the completion of the reaction time, the tube bomb was removed from the fluidized sand bath and cooled to room temperature by spraying with cold water. The gas in the tube bomb was vented and the contents were transferred quantitatively into a container using tetrahydrofuran (THF) as a solvent and subsequently Soxhlet extracted. The percent total coal conversion was calculated as follows:

$$\text{Percent Conversion} = 100 \left[ 1 - \frac{\text{dry, ash-free residue}}{\text{dry, ash-free coal}} \right]$$

Figure 24 shows a schematic diagram of the tubing

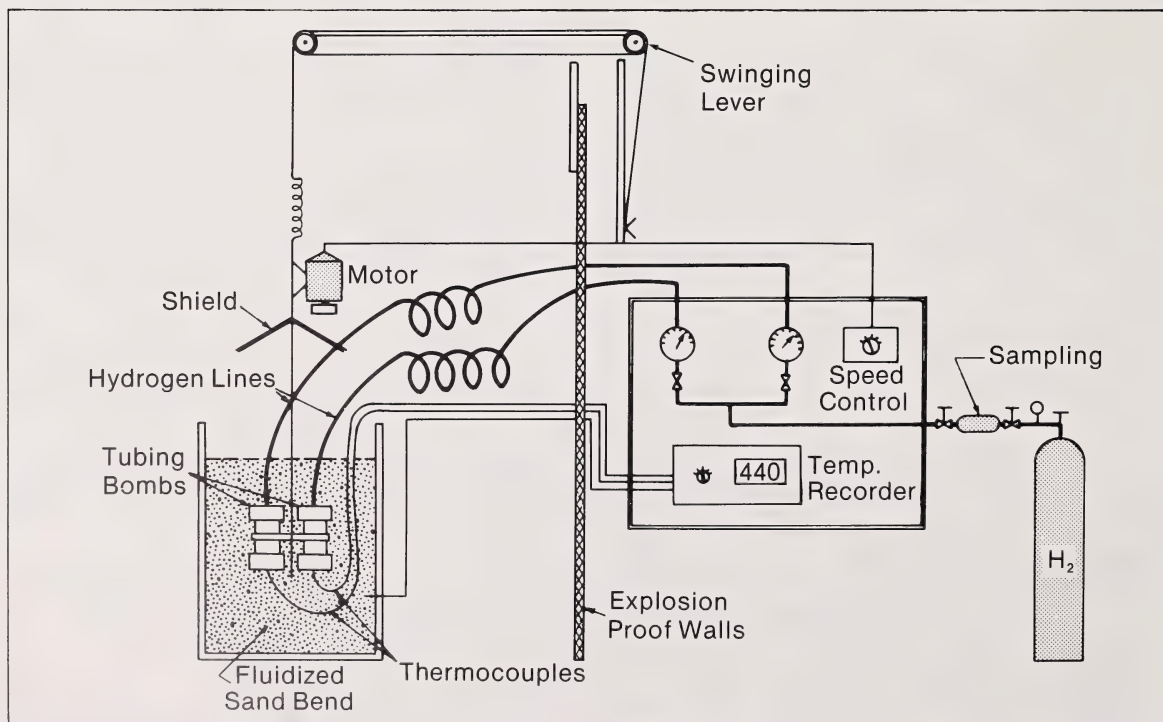


Figure 24. Schematic diagram of tubing bomb reactor for liquefaction studies

bomb reactor. The dependence of liquefaction behavior on the relative abundance of different macerals in three subbituminous coals and an American lignite (Texas) is shown in figures 25 and 26, respectively (Parkash *et al.*, 1983). An examination of the liquefaction data for all subbituminous and lignite coals tested indicated that solubilization susceptibility decreases with an increase in density fractions. Very likely, this correlation is due to the decrease in the combined huminite and liptinite contents — termed as “reactives” — in high density fractions. On plotting total conversion of parent coals and their density fractions against their huminite content, some linear correlations become apparent (figures 27 and 28). It appears to be much better for the Smoky Tower and Highvale subbituminous coals than for the Vesta samples. Similarly, the linear correlation is better for North Dakota lignite than for the Texas samples.

In the density fractions of the coals, the ratio of huminite to remaining macerals varies considerably from fraction to fraction. One can draw therefore, a tentative hypothesis that the contribution of the remaining macerals (semifusinite, fusinite, other inertinites and liptinite) to the solubility yield is minimal. However, an examination of the relationship between combined huminite-liptinite and percent conversion (figure 29) shows little difference from that displayed by huminite alone (figure 28), suggesting the reactive characteristic of liptinite macerals as well. Deviations from the regression line of figures 27-29 are, to a large extent, likely due to the variable reactivity of the semifusinite. Slight rank differences between the subbituminous and lignite coals may also play a part in the behavior of these coals. The reflectance values in table 24 indicate that the Texas coals are slightly higher in rank than those of North Dakota. To test this hypothesis, use was made of a formula first proposed by Guyot (1976) and quoted by Diessel (1979). This formula takes into account rank differences among coals as well as maceral composition. The relationship is expressed by the term “petrofactor” and is calculated by the following equation:

$$P (\text{petrofactor}) = \frac{\text{huminite reflectance}}{\text{reactive macerals } \%} \times 1000$$

where reactive macerals consist of huminite plus liptinite. Petrofactors were calculated for the parent lignites and their density fractions for which liquefaction data were available; the results were plotted against conversion yield in figure 30. A comparison of the correlation coefficients show that the relationship is not as good as that shown by figures 28 and 29 for huminite and huminite plus liptinite respectively (Parkash *et al.*, 1984).

The quality of liquids generated upon liquefaction of different fractions of subbituminous coals (table 28) indicates that portions relatively richer in inertinites yield high density fraction asphaltenic materials (Parkash *et al.*, 1983).

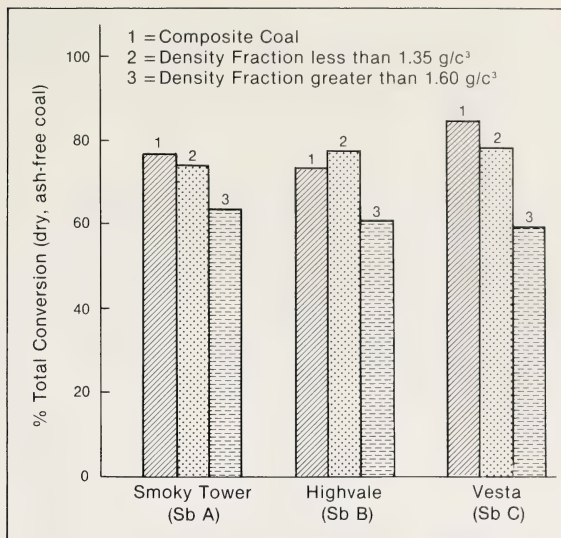


Figure 25. Dependence of conversion yields on density fractions of subbituminous coals

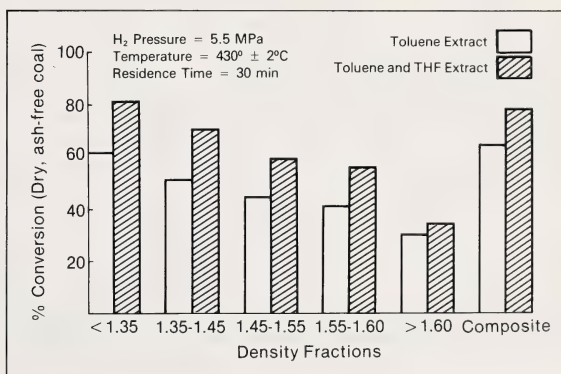


Figure 26. Dependence of conversion yields on density fractions of Texas lignite

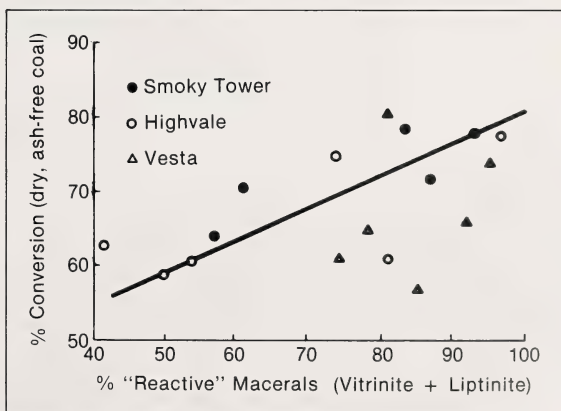


Figure 27. Dependence of conversion yields on “reactive” macerals

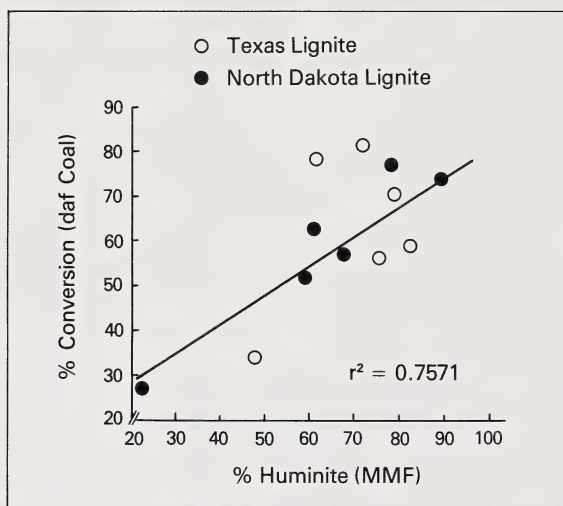


These data have provided some new insights into the role of different macerals in coal liquefaction in a semiquantitative manner, although it may not be possible to predict the liquefaction behavior of a coal from its maceral composition alone.

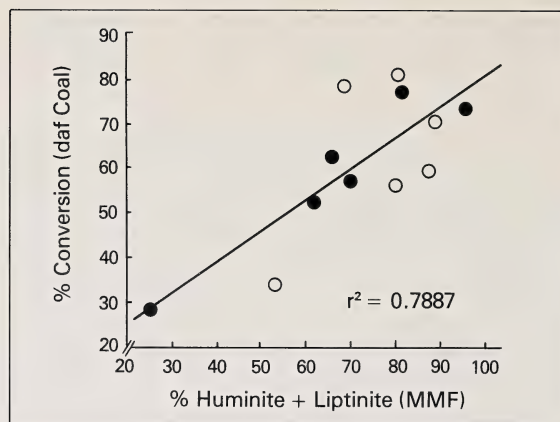
## Liquefaction reactivities of pure macerals and their admixtures

Table 29 presents the liquefaction conversion yields of handpicked macerals (greater than 90 percent purity) from the Vesta mine and their admixtures, under the I.G. Farben type process. The data indicate:

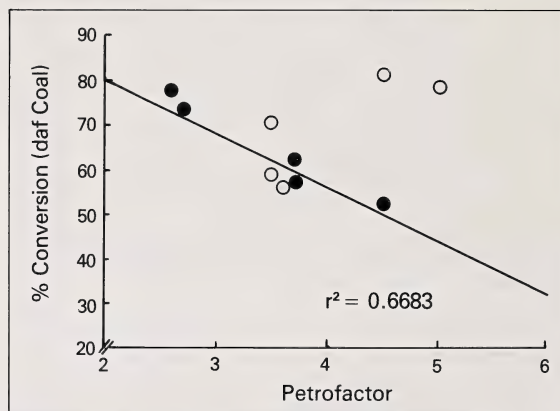
1. Conversions of 96 percent pure huminite and of coal



**Figure 28.** Dependence of conversion yields on huminite content of U.S. lignites and their density fractions



**Figure 29.** Dependence of conversion yields on combined huminite-liptinite contents of U.S. lignites and their density fractions



**Figure 30.** Dependence of conversion yields on petrofactors of U.S. lignites and their density fractions

**Table 28.** Quality of liquids\* produced from density fractions of Smoky Tower coal

	Percent vitrinite plus liptinite	Percent in THF solubles		
		Aromatics	Asphaltenes	Preasphaltenes
Density fraction:				
< 1.35 gcm <sup>-3</sup>	93.4	14.4	42.8	42.8
> 1.60 gcm <sup>-3</sup>	57.0	4.4	80.4	15.2
Recombined density fractions	75.2	9.8	73.5	16.7
Composite coal	76.4	2.9	82.2	14.9

\*Using thin-layer chromatography with flame ionization detection (TLC/FID).

**Table 29.** Reactivities of Vesta coal macerals for hydroliquefaction

Sample description	Percent conversion (daf basis)
Coal	19.8
Huminite (96% pure)	21.7
Fusinite (66% pure)	11.5
Huminite + liptinite (4:1)	33.8
Coal + huminite (3:1)	20.6
Coal + huminite (1:1)	28.6
Coal + huminite (1:3)	33.6
Coal + fusinite (1:1)	15.5
Liptinite + fusinite + huminite (1:2:5)	34.0

Process conditions: Hydrogen pressure = 1000 psi  
Sample/solvent (tetralin) ratio = 2:1  
Reaction temperature = 350°C  
Reaction time = 10 min.

Conversion was calculated on the basis of methylene chloride insolubles.

containing 73 percent huminite are almost identical. This fact suggests that the mineral matter content present in the coal compensates for the 23 percent difference in the huminitic content in the two samples. In other words, mineral matter in coal has a strong catalytic effect and, therefore, it may not be wise to predict the liquefaction reactivity of a coal entirely on the basis of its maceral composition without giving consideration to the content of mineral matter.

2. Conversion improves with the addition of extra

amounts of pure huminite in coal. This fact suggests the reactive role of huminite present in coal for liquefaction.

3. Diluting coal with fusinite results in lowering the conversion yields. This observation confirms the inertinitic behavior of fusinite in coal liquefaction.
4. Pure macerals (huminite, liptinite, fusinite), when mixed together mechanically in proportions normally present in Vesta coal, have a great synergistic effect in liquefaction.

## Petrography of liquefied residual materials

The studies which have been particularly rewarding in following the progressive alteration and resolidification of coal components during liquefaction are those carried out on residues obtained from autoclave batch tests performed over a range of temperatures (Kalkreuth and Chornet, 1982). It seems likely that, with the aid of optical microscopy, one may be able to predict with some confidence the outcome of hydrogenating a particular coal under specific conditions.

### Classification of residue components

The main categories of residue components derived from the liquefaction of coals are identified (plate 6).

#### A. Relatively unaltered organic components

These are macerals which have passed through the hydrogenation process with little or no morphological alteration. Identification is commonly easy because the features are similar to those of the original coal macerals, although reflectance is increased in many instances. Fragments of huminite in residues from subbituminous coal can still display a cell structure

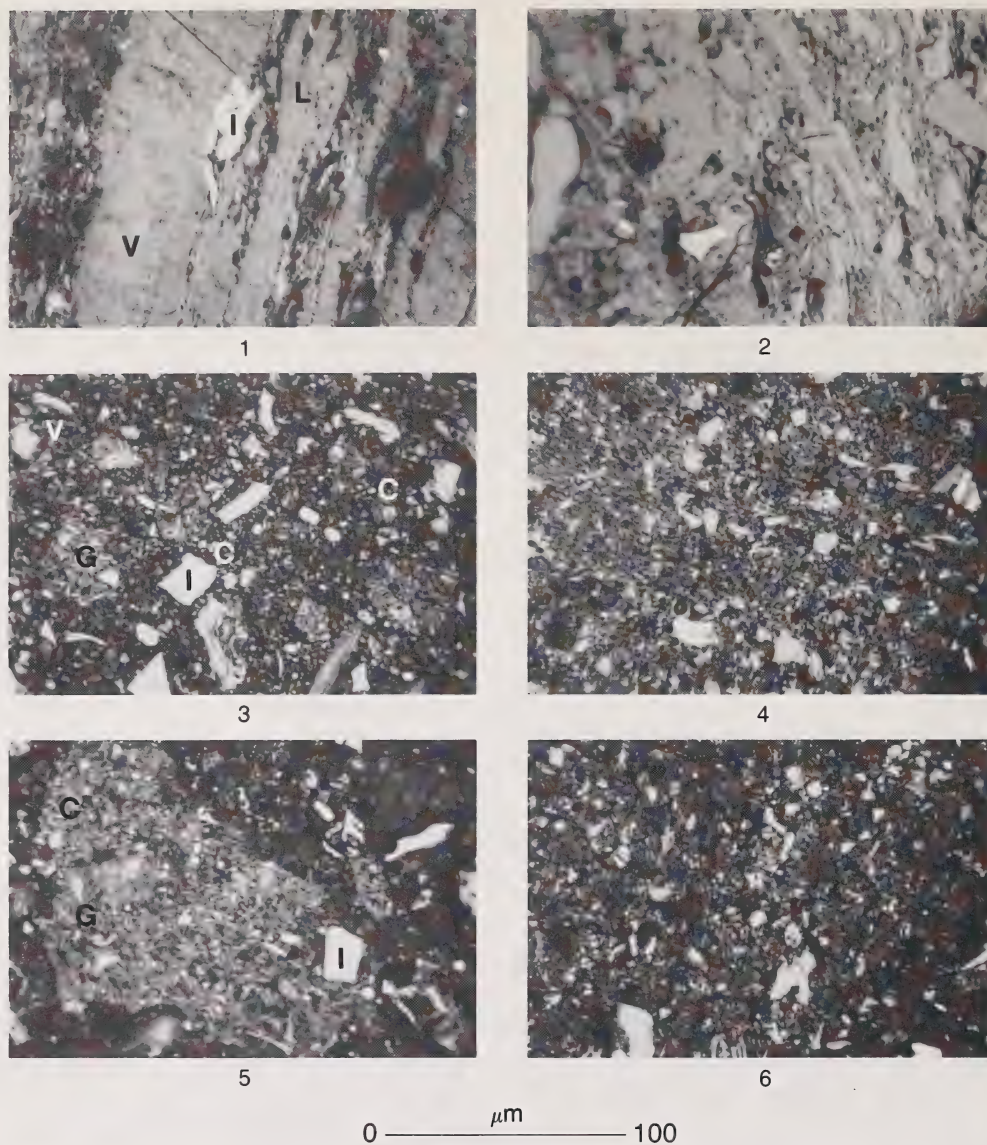
that enables the particular maceral precursor to be identified.

#### B. Thermally altered components

These are residue components of organic origin which have undergone a phase change. Their optical characteristics (reflectance, morphology) are different from those of the coal entities from which they have been derived. The materials and structures which constitute the plastic residue components of coal are vitroplast, cenospheres, semicoke and granular residue.

1. *Vitroplast*: when vitrinite from bituminous coals is heated, it may become plastic at a temperature of about 350°C. This component is termed "vitroplast". Vitroplast-like materials in the residues of subbituminous coals are believed to have been produced by the partial hydrogenation of huminite macerals, rather than through simple thermoplastic softening.
2. *Cenospheres*: cenospheres are globular bodies, often porous and with a reticulated internal texture. These pores may or may not be filled. Their development can take place as an immediate result





**Plate 6.** Photomicrographs of subbituminous coals and their residues after liquefaction:

(1) Smoky Tower coal, (2) Highvale coal, (3, 4) Residue from Farben type process - Smoky Tower and Highvale coals, (5, 6) Residue from CO-steam type process - Smoky Tower and Highvale coals showing huminites/vitrinite (V), vitroplast (V<sub>p</sub>), inertinite (I), coke/semicoke (C), granular residue (G)

of vitroplast formation. Cenospheres are believed to result from gas production by thermal cracking.

3. *Semicoke*: can form as a result of exposure to elevated temperatures of the solid products of liquefaction. In fact, semicoke may originate from semifusinite (nonplastic components), vitroplast and cenospheres.
4. *Granular residue*: submicron size material, the origin of which is not completely known at present. Much

of it may be derived from the physical dispersion of clay minerals intimately mixed with fine-grained inertinite.

### C. Inorganic components

Microscopically recognizable inorganic components present in liquefaction residues include:

- those which remain physically and chemically unaltered through the process, such as quartz,

- those which have undergone extensive physical breakdown during the process, such as clay minerals,
- those which have undergone major chemical and crystallographic transformations, such as conversion of pyrite to pyrrhotite,
- those minerals which are formed as a result of the process conditions, such as calcium carbonate, which crystallized in significant amounts during solvent-refined-coal production.

## Petrographic analysis of reaction residues

Table 30 gives the data from the petrographic analysis of residues from the liquefaction of the three Alberta subbituminous coals. Two processes were used, namely the I.G. Farben approach and the CO/steam process. Photomicrographs of the coals and their residues are shown in plate 6. The following are some of the general conclusions drawn from the optical examination of these materials.

1. Residual huminite has been changed to cenospheres, granular residue and semicoke.
2. Semifusinite has solubilized partially and the unreacted portion appears in the residue. During the liquefaction process large fragments of semifusinite have become finely comminuted and a large part of this fine material has been incorporated in the granular residue. Fusinite appears to have met a similar fate.
3. Unreacted or even partially reacted liptinite was not detected.
4. Mineral matter has apparently broken down to form fines and, together with fusinite and unreacted

semifusinite, constitutes the major part of the granular residue.

5. Comparison of residues from the two processes (I.G. Farben and CO/steam) showed:
  - in two of the three samples the I.G. Farben residue contained more semicoke,
  - although table 30 shows little pattern of variation in the amount of unreacted huminite between the two processes, the mode of occurrence is quite different; in the residue from the I.G. Farben process the huminite is dispersed uniformly while in the CO/steam residue it occurs in clusters,
  - reflectances measured on the huminite residue showed lower values in the CO/steam residue than in that from the I.G. Farben process. Presumably this situation is due to the higher process temperature in the former (400°C) as compared to the latter (460°C).

In conclusion, petrological methods are, no doubt, gaining ground as a means of characterizing the behavior of coal during hydrogenation; it is probably premature at present, however, to suggest that we now clearly understand the liquefaction behavior of a particular coal based on a comparative study of the petrographic composition of the raw coal.

The quantitative microscopic analysis of residue components may help in the evaluation of the partial reactivity of maceral constituents and in making a subjective comparison of the effectiveness of the different experimental conditions in the liquefaction processes.

The influence of the mineral matter content of a coal on its conversion potential is poorly understood at present. Strehlow and co-workers (1978) are of the opinion that the submicron-sized mineral components within huminite (vitrinite) may influence the conversion

**Table 30.** Petrographic analysis of liquefied coal residues

Composition of residues from various coals (percentage volume, mmf)	I.G. Farben process (P = 6.9 MPa; T = 460°C; t = 10 min)			CO/steam process (P = 5.5 MPa; T = 400°C; t = 10 min)		
	Smoky Tower coal	Highvale coal	Vesta coal	Smoky Tower coal	Highvale coal	Vesta coal
Huminite	13.4	12.1	9.6	10.4	21.8	8.9
Semifusinite	1.5	5.5	1.9	2.1	1.1	0.2
Fusinite	0.2	0.6	nil	1.1	0.6	nil
Granular residue*	72.8	71.6	75.8	80.5	67.9	83.2
Semicoke	3.1	6.0	7.8	4.0	4.1	1.0
Bright fragments	8.8	4.2	4.9	1.9	4.5	6.7
Cenospheres	0.2	nil	nil	nil	nil	nil
Average random reflectance of huminite (percent)	1.39	1.56	1.17	1.14	1.02	1.07

\*Particles less than 10  $\mu\text{m}$  which are not identifiable as individual components: includes both maceral and mineral-derived materials.



of coal into liquids. Their data show that the surface area of the submicron-sized mineral matter is significantly large and can be expected to induce a variety of catalytic reactions in coal liquefaction.

## Coal maceral morphology changes upon progressive hydrol liquefaction

In coal liquefaction, basic research on coal properties and their relationships to the amount and characteristics of process products has occupied a far less important place than research aimed toward specific engineering aspects of the processes themselves. One of the principal motivations in coal petrography is to identify important coal properties for new technologies such as coal liquefaction, so that predictive models eventually can be developed (Kuehn, 1982). Such models can be used to ensure quality control in a certain process as well as the evaluation of coals as potential feedstocks for that purpose. It must be recognized that previously no systematic research was undertaken to study the morphology and the nature of solid residues and to make comparisons with the petrography of the feed coal. The early work of Fisher and co-workers (1942) has shown that in coal liquefaction, variations in the liquid yields are greatly influenced by petrographic compositions. In an attempt to develop a classification system for subbituminous coal liquefaction residual materials, experiments were designed to study the morphology and the nature of solid residues upon progressive hydrol liquefaction of two Alberta subbituminous coals from the Horseshoe Canyon (Vesta) and Paskapoo (Highvale) geological formations at six different temperatures in the range 225° to 430°C at 10 minutes residence time.

The comparison of liquefaction conversion yields at different temperatures between two coals (Vesta and Highvale) shown in figure 31 indicates that at low temperatures the conversion from Highvale coal is somewhat better than from coal from the Vesta mine, but the difference narrows down at temperatures approaching optimum liquefaction yields (above 380°C). This observation probably reflects on the temperature susceptibilities for different subcomponents of reactive macerals (huminites and liptinites) present in these coals. This, of course, is quite expected, in view of the great degree of heterogeneity existing within a reactive maceral entity.

Plate 7 shows the microscopic morphology of the residual materials (methylene chloride insolubles) obtained from the short residence time (10 minutes) hydrol liquefaction of subbituminous coals at different temperatures.

Detailed examination of the residues under reflected light microscope have shown that:

1. Liptinite macerals do not participate at temperatures below 350°C and their morphology essentially remains unchanged. This is perhaps due to the fact that liptinitic macerals are insulated with huminites and thus fail to solubilize at lower temperatures.

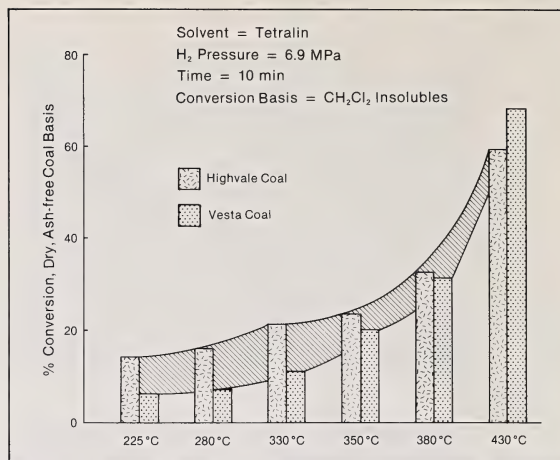
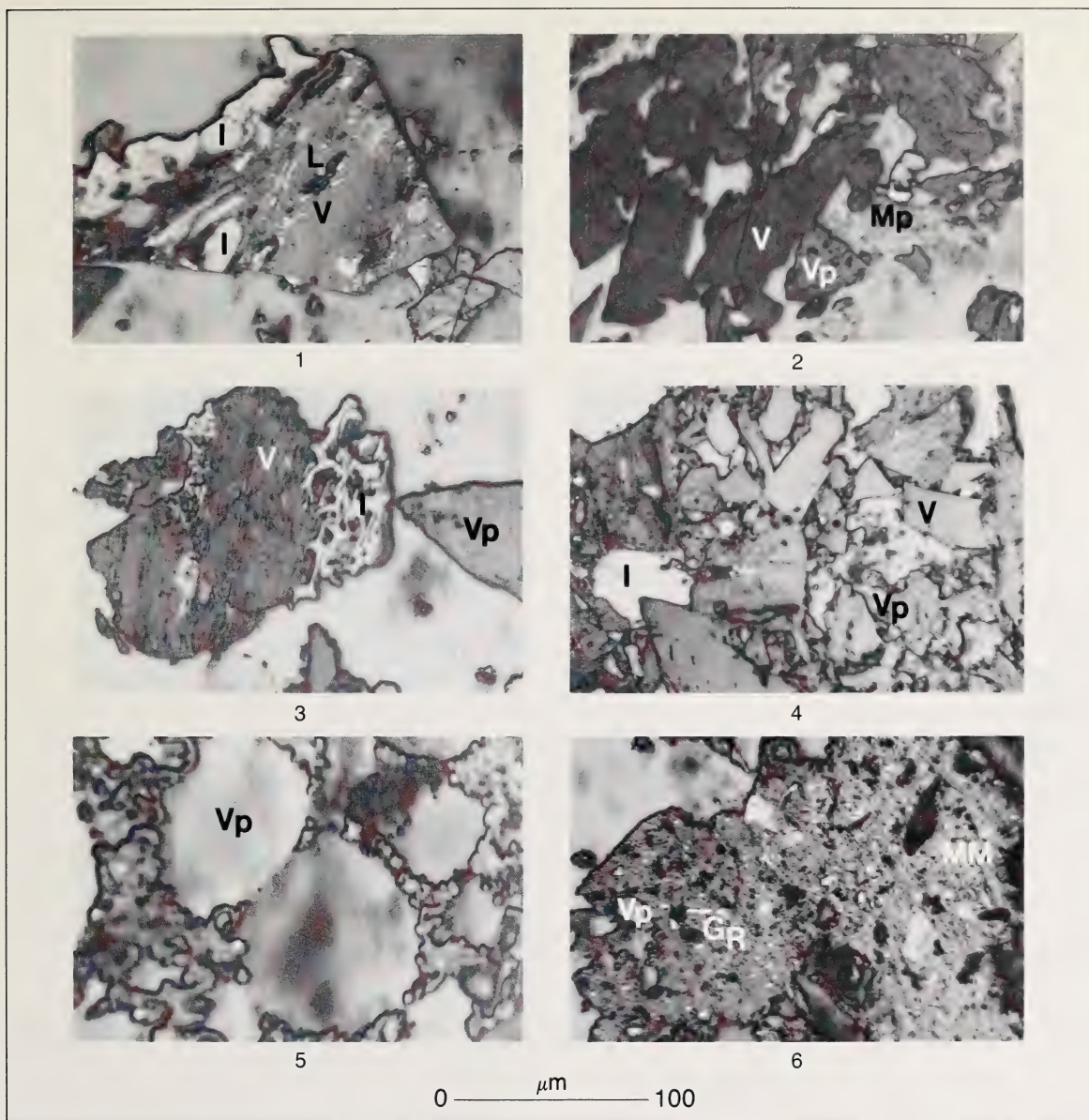


Figure 31. Liquefaction conversion yields at different temperatures

2. Above 350°C hydrogen enriched liptinitic macerals volatilize very rapidly and are solubilized.
3. In the presence of a hydrogen donor solvent (tetralin) the huminitic macerals gradually swell and become plastic at about 280°C.
4. The plastic material, called "vitroplast", is essentially a mixture of asphaltenes and preasphaltenes.
5. The swelling of huminites is closely related to changes in pore structure.
6. The porosity is increased by solvent at elevated temperatures, giving the residual coal sample a much larger total surface area than that of the parent coal.
7. Provided hydrogen availability is sufficient, upon subsequent hydrogenation the "vitroplast" is converted to liquid and gaseous products, leaving a network of coaly material which consists of cell wall derived material.
8. The degree of swelling of the inertinites in response to solvent treatment appears to be negligible when compared with that of huminites.
9. Inertinites, including low reflectance semifusinite, do not undergo any deformation at temperatures below 380°C.
10. Under optimum hydrogenation conditions (430°C) a portion of the semifusinite is converted to liquid and gaseous products while the rest becomes secondary semicoke (anisotropic structure).

Table 31 gives the petrographic composition of residues from liquefaction runs at different temperatures. When compared with the maceral composition of the feed coal, the data on liquefaction residues quantify the contribution of reactive macerals and demonstrate that liquefaction conversion increases with the consumption of macerals (table 32).

The reflectance distribution of a liquefied residual material from Highvale coal (figure 32) indicates that reflectance of macerals increases relative to the feed coal, and as a result, a distinction between semifusinite and fusinite cannot be obtained.



**Plate 7.** Photomicrographs of Vesta coal residues obtained upon progressive hydroliquefaction at different temperatures: (1) 225°C, (2) 280°C, (3) 330°C, (4) 350°C, (5) 380°C, (6) 430°C showing vitrinite (V), liptinite (L), inertinite (I), vitroplast (Vp), mesophase (Mp), granular residue (Gr), mineral matter (MM).

Another important observation in the microscopic examination of liquefied residual material is the occurrence of "hydrogenation rims" (plate 8). Detailed investigations indicate that, during liquefaction, hydrogenation (seen as "rims") starts from the surface of the particle and proceeds gradually towards the center. The hydrogenation rims are the reactive zones where oil, asphaltenes and preasphaltene-like materials, generated during coal liquefaction, are accumulated. The width of these zones increases

progressively from approximately 4 to 8 microns upon extraction in solvents of different polarity. This observation has enabled study of the solvent diffusion effect on solvolysis of huminitic macerals in subbituminous coals (Ozum *et al.*, 1985). Quasi-steady state and transient coal-solvent diffusion-chemical reaction models developed have indicated that solvent diffusion into the reactive maceral most likely is a rate limiting step in coal liquefaction.



**Table 31.** Petrographic composition of methylene chloride insolubles derived from Highvale coal liquefactions at different temperatures

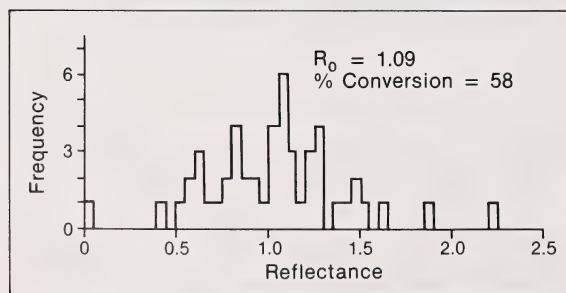
Liquefaction temperature (°C)	—	225	280	330	350	380	430
Petrographic entities	(volume percent)						
Huminite	69	62	51	42	12	2	—
Liptinite	4	4	4	4	3	—	—
Thermally altered huminite	—	2	10	14	41	38	12
Vitroplast	—	1	2	4	1	11	25
Mesophase	—	—	—	—	—	—	2
Inertinites	16	15	17	20	22	26	38
Granular particles	—	1	1	2	6	5	7
Mineral matter	11	15	15	14	15	18	26
Percent Reflectance	0.46	0.53	0.59	0.59	0.64	0.69	1.09
Percent conversion yield (daf basis)	—	14	16	21	23	32	58

**Table 32.** Liquefaction conversions versus reactive macerals consumption

Liquefaction temperature (°C)	225	280	330	350	380	430
Percent liquefaction conversion yield (daf coals basis)	14	16	21	23	32	58
Percent reactives in CH <sub>2</sub> Cl <sub>2</sub> insoluble residue	49	67	65	57	51	39
Percent consumption of reactives	5.5	8.2	12.3	21.9	30.1	46.6

Percent reactives in coals = 73

Calculation example:  $\frac{(73 - 69)}{73} \times 100 = 5.5$

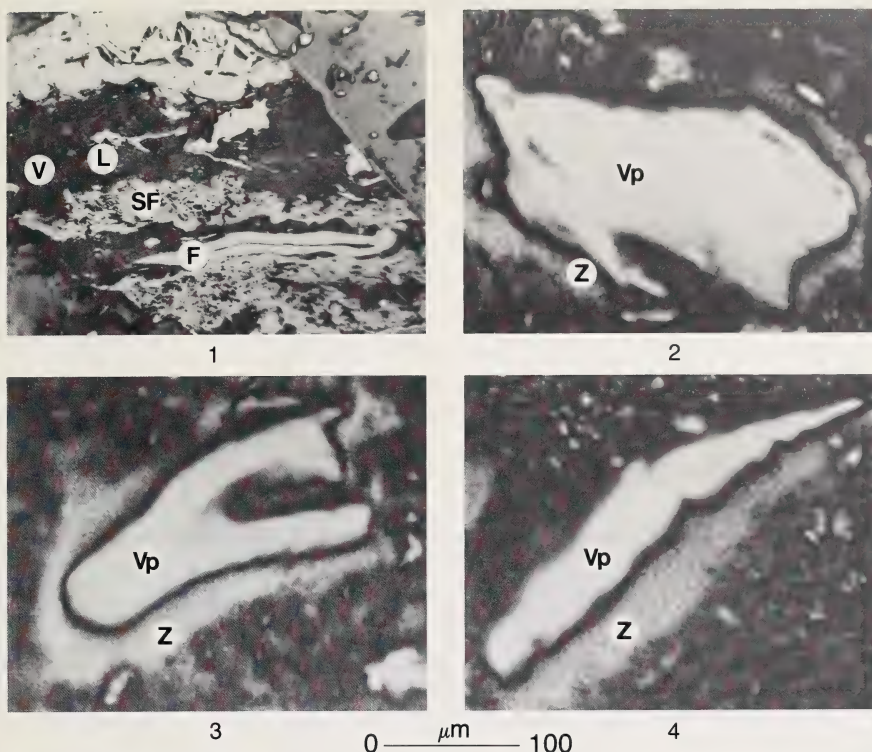
**Figure 32.** Typical reflectance distribution of liquefaction residue

These investigations have led to greater understanding of subbituminous coal liquefaction and have enabled to propose a very general petrographic classification of coal liquefaction residual materials

(table 33). The proposed classification is essentially based on three main categories of residue components as proposed by Mitchell and co-workers (1977): relatively unaltered organic components; thermally altered components (vitroplast, cenospheres, semicoke and granular residue); and inorganic components.

Extensive microscopic examination of residues from the liquefaction of three Alberta subbituminous coals using two processes (the I.G. Farben approach and the CO/steam process) and both batch autoclave tests and continuous runs (tables 34-37) suggests a possible path of alterations of huminitic macerals in liquefaction (figure 33). This path essentially confirms the findings on the characterization of coal liquefaction residues reported by American and Australian workers (Mitchell *et al.*, 1977; Shibaoka, 1982).

The effect of temperature on liquefaction of hand-picked huminite (98 percent purity) and inertinite (95



**Plate 8.** Photomicrographs of Highvale coal and liquefaction residues:

(1) Highvale coal, (2) Residue after hexane wash, (3) Residue after toluene wash, (4) Residue after THF-wash showing vitrinite (V), liptinite (L), semifusinite (SF), fusinite (F), vitroplast (Vp), and hydrogenation rim (Z).

**Table 33.** Classification of liquefied coal residue materials

Vitroplast	— Present as binder in the residue material
mesophase/anisotropic semicoke	— Occurs as a matrix in mineral matter rich carbonaceous residues
Isotropic semicoke	— Particles may have mosaic or non-mosaic texture; easy to differentiate as the former retains the original outline of the coal particles
Unaltered coal particles	— Shows characteristic features like cauliflower structure with high reflectance
Pyrolytic carbon	— Derived from original coal and catalyst
Mineral matter	

percent purity) is given in table 38. Liquefaction of inertinite occurred at somewhat higher temperature compared to that of huminite macerals which initiated between 350° and 400°C. The observation supported the findings reported by Shibaoka *et al.* (1984) for Australian coal macerals, where it was suggested that the major reaction pathway for the inertinite particles in the hydrogenation process appeared to be one of initial mild carbonization followed by hydrogenation.

The ease of liquefaction of huminite macerals in coal liquefaction compared to inertinite macerals of sub-bituminous and bituminous coals was recognized by several workers (Given *et al.*, 1975; Heng and Shibaoka, 1983; Parkash *et al.*, 1983b, 1984a). It was believed that huminite in the presence of a hydrogen

donor solvent (tetralin) first formed a plastic-like material called vitroplast, which subsequently was either converted to liquid and gaseous products or changed to mesophase and semicoke, depending on hydrogen availability within the system (Mitchell *et al.*, 1977; Shibaoka, 1981a, 1981b; Parkash *et al.*, 1984b). The inertinite, having a higher aromaticity (Pugmire *et al.*, 1984), was likely to have a large amount of reducible fused-ring aromatic units (King and Stock, 1982) and thus the demand for hydrogen was higher for inertinite during liquefaction (King *et al.*, 1984).

Whether comparative studies of maceral composition and rank with those of the relevant hydrogenation residues shall enable conversion rates and efficiencies to be assessed for the various coals has yet to be con-



**Table 34.** Petrographic composition of residues from liquefaction of Judy Creek coal using different processes

Process description	P=1750 psi (H <sub>2</sub> ) T=470°C t=0 min Solvent=tetralin Fe <sub>2</sub> O <sub>3</sub> catalyst	P=1000 psi (CO) T=400°C t=30 min Solvent=tetralin K <sub>2</sub> CO <sub>3</sub> additive	P=1500 psi (H <sub>2</sub> ) T=430°C t=30 min Solvent=tetralin K <sub>2</sub> MoO <sub>4</sub> catalyst	P=1500 psi (H <sub>2</sub> ) T=400°C t=60 min Solvent=Suncor bitumen Fe <sub>2</sub> O <sub>3</sub> catalyst	P=1000 psi (CO) T=400°C t=60 min Solvent=Suncor bitumen K <sub>2</sub> CO <sub>3</sub> additive
<b>Petrographic components (volume percent)</b>					
Altered huminite	20.3	29.6	16.0	50.5	14.3
Cenosphere	—	0.5	2.0	1.0	—
Inertinite	1.0	2.6	1.3	7.6	3.0
Mesophase	—	—	—	—	—
Coke	—	1.5	2.6	1.0	0.7
Granular residue	63.7	48.5	52.5	20.3	45.3
Mineral matter	15.0	17.3	25.6	19.6	36.7

**Table 35.** Petrographic composition of residues from liquefaction of Fox Creek coal using different processes

Process description	P=1000 psi (CO) T=400°C t=30 min Solvent=tetralin	P=1500 psi (H <sub>2</sub> ) T=430°C t=30 min Solvent=tetralin	P=1500 psi (H <sub>2</sub> ) T=400°C t=60 min Solvent=Suncor bitumen	P=1000 psi (CO) T=400°C t=60 min Solvent=Suncor bitumen
<b>Petrographic Components (volume percent)</b>				
Altered huminite	24.7	22.7	24.4	26.3
Cenosphere	—	—	—	—
Fusinite	4.0	9.0	3.3	5.0
Mesophase	—	2.7	—	—
Coke	1.6	4.0	6.7	0.3
Granular residue	40.0	25.3	36.3	35.3
Mineral matter	29.7	36.3	29.3	33.0

**Table 36.** Petrographic analysis of Judy Creek and Fox Creek residues  
(mini-autoclave liquefaction experiments)

Maceral	I.G. Farben type process <sup>1</sup>		CO/steam type process <sup>2</sup>	
	Fox Creek (volume percent)	Judy Creek (volume percent)	Fox Creek (volume percent)	Judy Creek (volume percent)
Unaltered huminite	0.3	—	0.3	—
Altered huminite	10.0	1.7	4.3	6.0
Granular residue	80.0	85.0	63.7	63.3
Fusinite	2.7	4.0	9.7	9.3
Mesophase	2.7	0.6	8.7	5.0
Coke	—	—	—	0.4
Mineral matter	4.3	8.7	13.3	16.0

1: THF insoluble residues using anthracene oil as solvent with hydrogen gas.

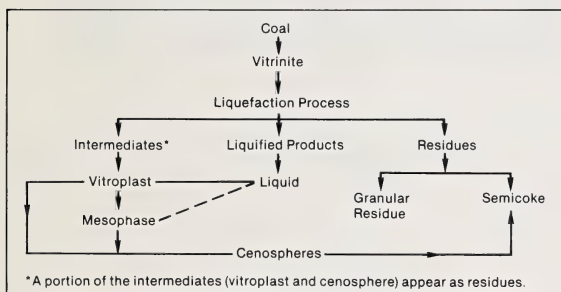
2: THF insoluble residues using anthracene oil and CO gas.

**Table 37.** Petrographic composition of solid residues derived from hydroliquefaction of Highvale coal from continuous bench unit runs

Petrographic entities (percent)	#1	#2
Average reflectance of residue	2.12	1.87
Undissolved material		
Huminites	7	6
Liptinites	1	0
Inertinites	22	24
Mineral matter	47	48
Solvent affected material		
Partially dissolved coal	2	2
Vitroplast (coagulant)	5	8
Amorphous material (asphaltene)	12	8
Heat affected material		
Mesophase/semicoke	2	2
Pyrolytic carbon	2	2

Note: #1 - H/coal ratio = 1.2 m<sup>3</sup>/kg; 455-470°C

#2 - H/coal ratio = 1.6 m<sup>3</sup>/kg; 440-450°C

**Figure 33.** Possible alterations of huminite/vitrinite macerals in liquefaction

firmed. For the assessment of coal hydrogenation performance the use of microscope techniques may not be very efficient. It is essential to pool results from both chemical and microscopic investigations to derive a better understanding of the hydroliquefaction process.

## Petrographic composition of chars

The second most important experimental and scale-up program at the Alberta Research Council is the testing of subbituminous coals for their pyrolysis behavior. The pyrolysis of coal yields relatively hydrogen-rich volatiles but the major byproduct is the relatively

**Table 38.** Petrographic composition of chars from Vesta (subbituminous C) coal

Charring temperature (°C)	550	650	700	800
Petrographic components	(volume percent)			
Unaltered huminite	1.0	—	—	0.7
Altered huminite	80.4	81.0	87.7	85.7
Fusinite	1.3	1.4	0.7	0.3
Granular residue	8.0	9.3	6.6	9.0
Coke	—	—	—	—
Mineral matter	9.3	8.3	5.0	4.3
Percent reflectance	1.38	1.84	2.03	2.82
Percent char yield (coal)	61.2	48.4	44.6	43.1



carbon-rich solid char which can be utilized profitably in thermal power generation. In an attempt to understand whether the optical characterization of chars contributes to an understanding of the role of different macerals in their reactivities, the petrographic composition of chars produced by flash pyrolysis/hydropyrolysis from subbituminous coals under different sets of conditions were determined. Data for some of the chars produced are recorded in tables 38-41 and their photomicrographs are shown in plate 9. In the petrographic analysis for chars, the classification system used for various petrographic entities is the same as that proposed for coal liquefaction residual materials. Reflectance distributions of coals and their chars produced under different sets of conditions are shown in figures 34 and 35. Among the important observations of general interest in char

characterization are the following:

1. Huminite participates more readily, fusinite remains unchanged.
2. Most of the huminite is simply thermally altered and is not consumed under experimental conditions of pyrolysis.
3. At an early stage in pyrolysis at lower temperatures a thermoplastic like material is formed from huminite.
4. The degree of thermoplasticity of huminite is related to changes in pore structure.
5. The periodic constrictions in the pores, present in the feed coal, are removed during pyrolysis.
6. New micropores are generated and the macropore structure expands.
7. The porosity is increased, giving the char a much larger surface area than the feed coal.

**Table 39.** Petrographic analysis of chars 550°C (mini-pyrolysis unit)

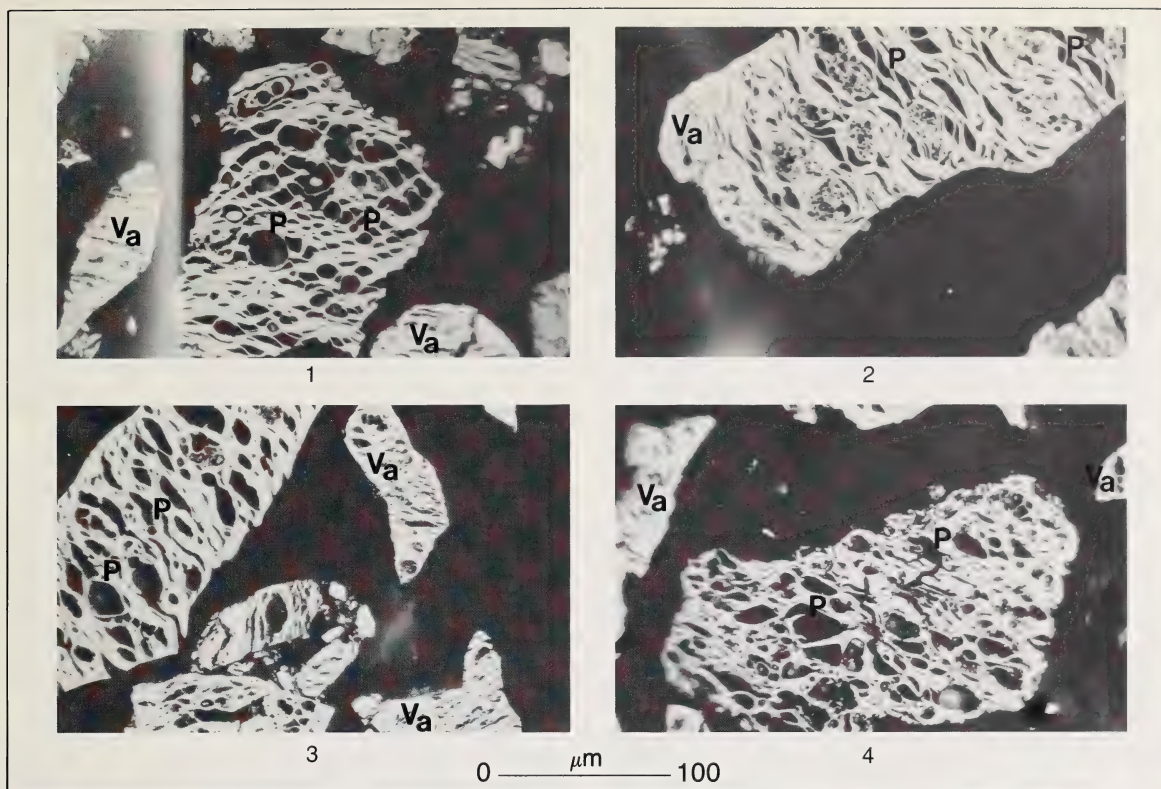
Maceral (volume percent)	Coal		
	Blackfoot	Bow City	Highvale
Humoplast (vitroplast)	6.7	8.7	—
Altered huminite	82.0	59.3	68.3
Granular residue	2.3	5.3	5.4
Fusinite	4.0	4.3	16.3
Coke	—	—	—
Mineral matter	5.0	22.3	9.7
Mesophase	—	—	0.3

**Table 40.** Petrographic analysis of chars 700°C (mini-pyrolysis unit)

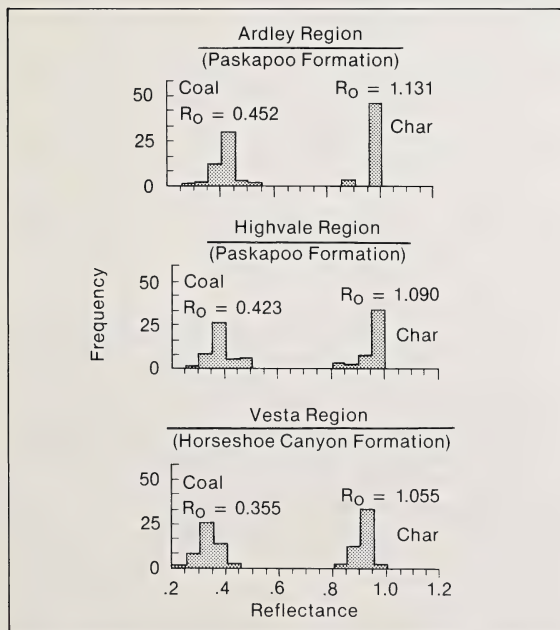
Maceral (volume percent)	Coal		
	Blackfoot	Bow City	Highvale
Altered huminite	80.7	75.0	78.0
Granular residue	3.3	5.0	3.3
Fusinite	3.0	2.7	7.3
Coke	—	0.6	0.3
Mineral matter	13.0	16.7	11.0

**Table 41.** Petrographic analysis of chars (bench pyrolysis unit)

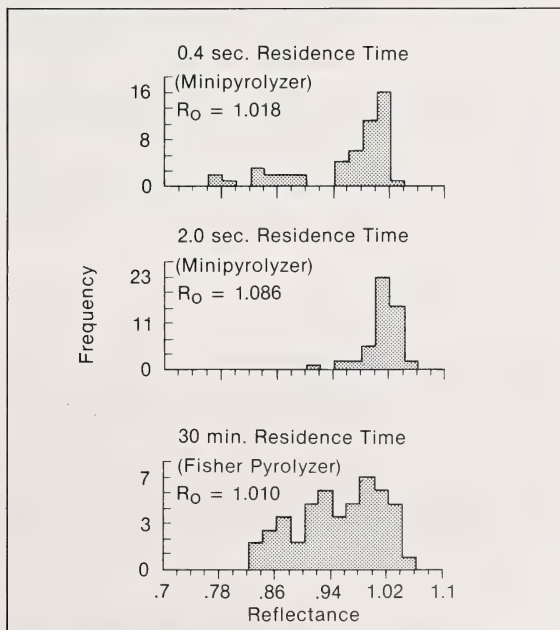
Maceral (volume percent)	Highvale 700°C, 800 psi 1.67 sec residence time	Vesta 700°C, 800 psi 1.8 sec residence time
Altered huminite	83.0	80.3
Granular residue	9.3	7.7
Fusinite plus inertinites	2.3	3.7
Mineral matter	5.3	8.3



**Plate 9.** Photomicrographs of chars from Vesta coal showing thermally altered vitrinite ( $V_a$ ) and pores formed (P) (1) 550°C, (2) 650°C, (3) 700°C, (4) 800°C



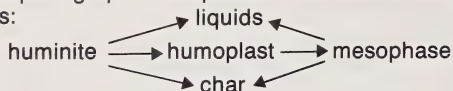
**Figure 34.** Reflectance distribution of coals and their 700°C chars produced in the Fisher analyser



**Figure 35.** Reflectograms for Highvale chars prepared under different process conditions at 700°C



The main reactions involved in pyrolysis in relation to the petrographic composition can best be summed up as:



From the data available at present, it would be premature to draw any conclusions of practical significance for a technical process like coal pyrolysis.

## Conclusions and comments

The petrographic characteristics of the subbituminous coals of the Alberta plains (previously almost unknown in contrast to information available on their chemical composition) have been extensively investigated. The data in this report should serve as a useful source of information in developing future applications — academic and applied — for explaining, improving and controlling the technological behavior of Alberta's large reserves of subbituminous coals. On the average, the huminite content in coals from the Horseshoe Canyon Formation (88 percent) is higher in comparison to coals from the Paskapoo Formation (72 percent). Reflectance measurements show a range of 0.36 to 0.47 with a rough correlation to the ASTM rank designations of these coals. Microscopic examination of liquefaction residual materials indicate total conversion of the liptinite, extensive conversion of huminite and possible partial conversion and reactivity of the semifusinite maceral of the inertinite group. Examination of the relationship between liquefaction yields and petrographic composition shows only a correlation with huminite-liptinite content. Though these investigations have not provided a model that can be used to predict liquefaction yields from a given coal based on its maceral composition and rank, nevertheless substantial information of an academic nature has been gathered on the morphology and reactivity of coal macerals which should be beneficial in understanding the chemistry of the subbituminous coals of the Alberta plains. For the assessment of coal hydrogenation performance, the use of microscope techniques may not be very efficient and it is essential to pool results from both chemical and microscopic investigations to derive a better understanding of the hydroliquefaction process.

### Comments

The petrographic analysis of coal is more of an art than a science. The problems involved in the identification of low rank coal macerals are compounded when those materials have undergone phase changes, severe comminution and increase in the level of reflectance.

Microscopic examinations of coals and of residues from hydrogenation runs, though very useful in following the reactivity behavior of several of the important

coal macerals, are not sufficient for a thorough understanding of what happens in liquefaction processes. Several of the residue classes are transitional and it is difficult to decide precisely where to set the thresholds between coal liquefaction residual materials. The sequence vitrinite (huminite) — vitroplast — cenospheres — semicoke poses particular problems.

Even with present limited knowledge on the role of coal macerals in liquefaction, petrography of residue components does provide some clear ideas as to what components should and should not be present in the residues following optimum conditions of liquefaction. The technique, therefore, has potential as a means of diagnosing some problems or inefficiencies in operation. For example, excessive proportions of unreacted huminite (vitrinite), vitroplast or semicoke could indicate that conditions were not optimal; on the other hand, large proportions of fusinite, semifusinite, or granular residue might indicate even less efficient operation. Much more needs to be done about the relative behaviors of coal macerals in different technical processes. The liquefaction behavior of a coal has been shown to be influenced by (among other things) the way in which the reactive macerals and inertinites are associated with each other in the coal. This fact, together with the very variable properties of semifusinite, necessitates a reassessment of the role of inertinite in a technical process. At present, the use of petrography to predict with some confidence the outcome of hydrogenating a particular coal under specific conditions is still in the developmental stage. One may need to move away from traditional procedures of petrography and find new approaches (perhaps the use of automatic image analysis) which would enhance ability to predict coal reactivity behavior with greater accuracy.

As for immediate research interests, petrography may have an appropriate impact in the investigations of an array of problems that commonly crop up with the utilization of certain coals in combustion. A major barrier to the optimal utilization of coals and pyrolysed fuel solids in combustion has been a general lack of definitive data on the influence of their maceral and mineral constituents on process performance. It should be worthwhile to devote some efforts in this direction.

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